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A REVIEW OF HAZARD ASSESSMENT PROCEDURES FOR LIQUID GUN PROPELLANTS

Prepared by

University of Arkansas
Fayetteville, Arkansas 72701

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US ARMY BALLISTIC RESEARCH LABORATORY
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Safety testing procedures for energetic liquids are reviewed. A rationale for development of a protocol for safety testing of liquid gun propellants is suggested. A compilation of safety test data for gun propellants, along with comparison data for explosives and rocket propellants, is presented. Applicability of test methods developed primarily for explosives and high energy rocket propellants to the assessment of hazards that may be encountered in handling, storage, and transportation of gun

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propellants is considered. Test procedures which should be further evaluated are identified. In general, the data available on the response of liquid gun propellants to these test procedures are not sufficient for a thorough evaluation of the tests' usefulness for assessing liquid gun propellant hazards. Additional work is recommended to provide test data which can be used to compare the response of gun propellants with other energetic liquids for which there is an extensive safety experience data base. Further work is also recommended to characterize the energy input and boundary conditions for the selected test procedures to provide a means for evaluating the tests with respect to severity and correspondence to conditions which may be encountered in handling, storage, and transportation.

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I. INTRODUCTION

Liquid gun propellant evaluation programs are developing requirements for candidate test propellants in larger than laboratory-scale quantities. Methodology is needed for assessment of hazards that may be encountered in handling, storage, transportation, and end use. There are numerous test procedures applicable to liquid gun propellant safety testing, but it cannot be assumed a priori that procedures developed for other classes of materials (e.g., rocket propellants or explosives) are equally applicable to liquid gun propellants. For example, a test procedure developed to quantify the shock sensitivity of high energy rocket propellants may not identify a lower, but nevertheless important, degree of shock sensitivity of a candidate liquid gun propellant.

The purpose of this work was to make an initial assessment of the hazardous material safety testing procedures that have been applied to energetic liquids and to provide recommendations for additional test requirements which may be indicated for assessing liquid gun propellant hazards. Three subordinate tasks were involved. First, a review of selected classified literature relating to liquid gun propellant development and testing was conducted at the U.S. Army Ballistic Research Laboratory. Second, a limited review was made of the published scientific literature in pertinent areas, particularly in detonation physics. Third, visits were made to selected government and private organizations involved in the development and testing of liquid gun propellants. These visits were for the purpose of discussing unpublished and on-going work pertaining to safety testing of liquid gun propellants.

In Part II a discussion of tests which have been used to assess hazards of energetic liquids is presented. Emphasis is placed on test procedures that are thought to be of highest priority for evaluation. Test methodology, typical test results, observations of test variability, and test interpretation are discussed. Part III is a summary of test results reviewed which pertain to hazard evaluation of liquid gun propellants. In Part IV the reported test results are discussed and recommendations are made for future test programs.

II. SAFETY TESTING OF LIQUID MATERIALS FOR POTENTIAL EXPLOSIVE HAZARD

The utility of explosives and propellants derives from their potential for rapid chemical reaction with attendant energy release. A propellant (in contrast to an explosive) is designed to release energy in a controlled way, as in a rocket motor or a liquid gun propellant. The rate of energy release for a chemical reaction depends on external conditions as well as the chemical structure of the material. Hence, a propellant may, under conditions different from those of its intended use, release energy at a rate sufficient to cause destruction. Materials useful as gun propellants are a case in point.

The "safety" of any potentially explosive material relates to its propensity for uncontrolled burning or explosion resulting from externally imposed conditions. Such a definition of the term "safe" extends

beyond the actual propellant application conditions in a gun to storage, handling,and transportation. The material should exhibit highly repeatable burning characteristics under design usage conditions but should not react violently in response to external stimuli from handling, storage,and transportation. Ideally, it should not react violently even to external conditions which may arise under abnormal conditions, such as accidents. In this regard, safety requirements imposed for propellant (and explosives) handling, storage and transportation inevitably involve compromises. It cannot reasonably be expected that such materials can exhibit all of the desired in-use features (for example, ease of ignition in use) and all of the desired safety features (for example, relative difficulty of ignition under all accident conditions).

However, the in-use and safety requirements of propellants and explosives are not mutually exclusive. The development of commercial explosives to their present status affirms this fact. Furthermore, the logic to be followed in selection of propellant materials which perform as desired in use, yet are safe, is deceptively simple:

1. Characterize the conditions imposed on the material in use, for example, in a gun.

2. Characterize the conditions which may be imposed on the material in handling, storage, and transportation.

3. Characterize the reaction proces. of the material as a function of the conditions identified in steps 1 and 2.

4. Select a material that satisfies the performance criteria of the gun but does not react violently to the conditions of handling, storage, and transportation (and to the extent possible to conditions resulting from accidents in handling, storage, and transportation).

The present process of selection of candidate materials for use as propellants does not follow this recipe, for the following reasons:

1. Actual performance (e.g., gun performance) cannot be completely predicted, again due primarily to our inability to predict propellant burning characteristics under gun operating conditions. The approach is to test the material under actual firing conditions to ascertain performance and safety.

2. We are not able to specify accurately the conditions that may be imposed on a material during normal handling, storage, and transportation; and we know less about the conditions that may be encountered in accidents. The approach is usually to subject the material to external energy inputs which are considered to be at least as "severe" as those expected in handling, storage, and transportation.

In general, the safety criteria for a propellant material relate directly to the response of the material to inputs of energy. Such inputs of energy, although theoretically reducible to a common thermal energy input

basis, are usually (somewhat arbitrarily) classified in separate categories as follows:

1. Thermal input (heat transferred into material);

2. Impact energy input;

3. Shock energy input;

4. Electrical discharge energy input.

In this literature review, emphasis was placed on the first three hazard evaluation categories listed above. No references were found on direct electrical discharge energy input measurements on liquid gun propellants, except data obtained in gun performance ignition studies.

The last three energy input categories can all be considered as forms of work-energy inputs (in the thermodynamic sense) which are converted locally in the material to thermal energy. The quantitative description of the conversion of such work-energy inputs to thermal energy is the province of irreversible thermodynamics; we do not as yet know how to compare the different types of energy inputs on a common basis. Hence, a test protocol usually includes multiple test procedures to determine the response of materials to each of these categories of input energy.

A. Thermal Energy Input Tests

This type of test may be directed to the ease of ignition of volatiles produced by the material or to the material's stability at increased temperature. In ignition tests, which are primarily directed to determination of fire hazard, the temperature of the material is determined at which it produces volatiles sufficient to allow piloted ignition in the gas phase above the material, or at which the material (or its gaseous products) spontaneously ignites. Thermal stability tests are usually directed to the determination of the maximum temperature below which the material does not generate reaction heat at a rate greater than that which can be transferred to the surroundings. If this temperature is exceeded for a material which can undergo an exothermic reaction, the temperature will increase uncontrollably and a "thermal explosion" will result.

Thermal Explosion Theory, introduced by Semenov and Frank-Kamenetskii,[1,2] provides a rationale for understanding and correlation of the response of energetic materials to thermal stimulii. Application of the energy balance principle to a homogeneous, isotropic, heat-generating material in which heat transfer is limited to conduction gives the differential equation for the temperature distribution in the material as a function of time:

1. N. N. Semenov, Chemical Kinetics and Chain Reactions, Oxford University Press, London (1935).

2. D. A. Frank-Kamenetskii, Diffusion and Heat Exchange in Chemical Kinetics, Princeton University Press, Princeton, NJ (1955).

$$\rho C \frac{dT}{dt} = \lambda \nabla^2 T + \rho Q \quad (1)$$

Energy Accumulation Rate	=	Energy Transfer Rate (Net)	+	Energy (Thermal) Production Rate

where T = local temperature
t = time
ρ = local density
C = local heat capacity
λ = local thermal conductivity
Q = thermal energy production rate per unit mass (from chemical reaction)

The general solution to this second order partial differential equation involves two arbitrary constants whose values depend on the initial and boundary conditions imposed on the material. Because of the dependence of the temperature on such boundary conditions, the temperature at which a material can dispose of the heat produced internally from chemical reaction as fast as it is being produced is not a unique value. This temperature depends on the transport properties of the material and on the boundary conditions (primarily heat transfer boundary conditions). Consequently thermal stability values (temperatures) obtained by different experimental techniques may not be directly comparable. In principle, the effect of non-thermal energy inputs into a material, to be discussed subsequently, can also be treated via the thermal explosion theory if the conversion rate of such energy inputs to thermal energy can be quantified. Unfortunately, information in this area is almost totally lacking.

There are a large number of test procedures which have been used to estimate "safe handling/storage" temperatures. As a class they are similar in that thermal energy is transferred to the material at a specified rate and the temperature at which the material gives evidence of reaction is noted. The rate of heat input varies greatly with the test procedure. A literature search revealed four sources of data for response of liquid gun propellants to controlled thermal energy inputs. These data were derived from conventional flash-point and ignition temperature tests and differential thermal analyses, from "thermal surge" tests developed at the Naval Ordnance Laboratory, and from variations of the "thermal stability" tests developed by the Interagency Chemical Rocket Propulsion Group (ICRPG). Some data on the response of containerized materials to fire exposure, referred to as "bonfire" tests, are also noted, although they are considered of little value for quantitative evaluation purposes. These test procedures will be described briefly.

1. Ignition Temperature. The objective of this test is the determination of the lowest temperature at which vapors from the material will spontaneously ignite in air. The result can be expected to depend on geometry of the vapor/air mixture sample even for a homogeneous gas/air mixture. The (Setchkin) Autoignition Temperature Test, standardized as

ASTM D286-36, has been widely applied to this type of measurement for a large number of materials. The experimental apparatus consists of a one-liter spherical flask maintained at a constant temperature. A liquid sample is injected into the flask and the time to ignition (determined by the appearance of a flash) is recorded. The test is repeated at higher and/or lower temperatures, as indicated; and the ignition time is determined as a function of temperature. The (extrapolated) temperature at which the ignition time becomes "infinite" is the autoignition temperature. Although an initial sample size of 0.05 ml (liquid) is usually prescribed, tests are repeated for different sample volumes to determine the minimum value of the ignition temperature.

2. Flash Point. The objective of the flash point test is the determination of the lowest temperature at which the material evaporates rapidly enough to form a flammable vapor/air mixture over the liquid surface. Because the formation of a flammable mixture in the vapor space depends on the evaporation of the liquid and its subsequent mixing with air, the test result is expected to be dependent on the sample and test chamber geometry. Methods have been standardized for open and closed container test procedures (ASTM 92-72 Cleveland Open Cup Flash Point Test and ASTM TAG Closed Cup Flash Point Test). In either case a small pilot flame is passed over the liquid surface, or at a designated opening where the sample vapors exit, at intervals of increasing temperature. The lowest liquid temperature at which the application of the pilot flame causes the vapors above the surface of the liquid to ignite is taken as the flash point.

3. Differential Thermal Analysis (DTA). This technique is based on measurement of the difference of internal energies, or heat contents, between an inert reference material and the sample material when both are heated in a similar thermal environment. Usually, the two materials are simultaneously exposed to a thermal environment which produces a linear temperature increase of the reference material. Due to the limitations (difficulties) in achieving accurate and reproducible high rates of heat transfer to the test sample in conventional DTA apparatus, sample temperature increase rates are typically low, i.e. less than 40°C/min. Differential thermal analysis measurements of the temperature at which an exothermic reaction is first observed is indicative of the thermal stability of the material.

4. Thermal Surge. This test procedure, developed at the Naval Ordnance Laboratory, is designed to determine the response of small, highly confined samples to very rapid heating (μsec to a few msec) to temperatures to 1000°C. A 2.1 μL sample is enclosed in a 6.35-cm length of stainless steel hypodermic needle tubing. The tubing is heated very rapidly by discharging a capacitor through it, and its resistance is measured as a function of time. The temperature of the tubing is determined from separate measurements of its resistance at different known temperatures. Explosion of the sample is evidenced by an abrupt change in the electrical resistance when the tube wall bursts. Hence, the temperature of the sample container and the delay time before explosion are determined by measuring the resistance of the hypodermic needle tubing as a function of time. The delay time to explosion is measured with an electronic timer which is started by

a signal from the capacitor discharge and stopped by a signal from a microphone located near the bursting sample tube. The apparatus is described by Kendall and Rosen[3] and Wenograd.[4] Typical data presented as plots of delay time to explosion against reciprocal temperature are shown in Figure II-1 from Kendall and Rosen[3] and Stull.[5] The delay time can be related to frequency factors and activation energies for describing the reaction kinetics and is a measure of the rate of reaction (rate of energy release) and hence, sensitivity of the material to intense thermal energy input.

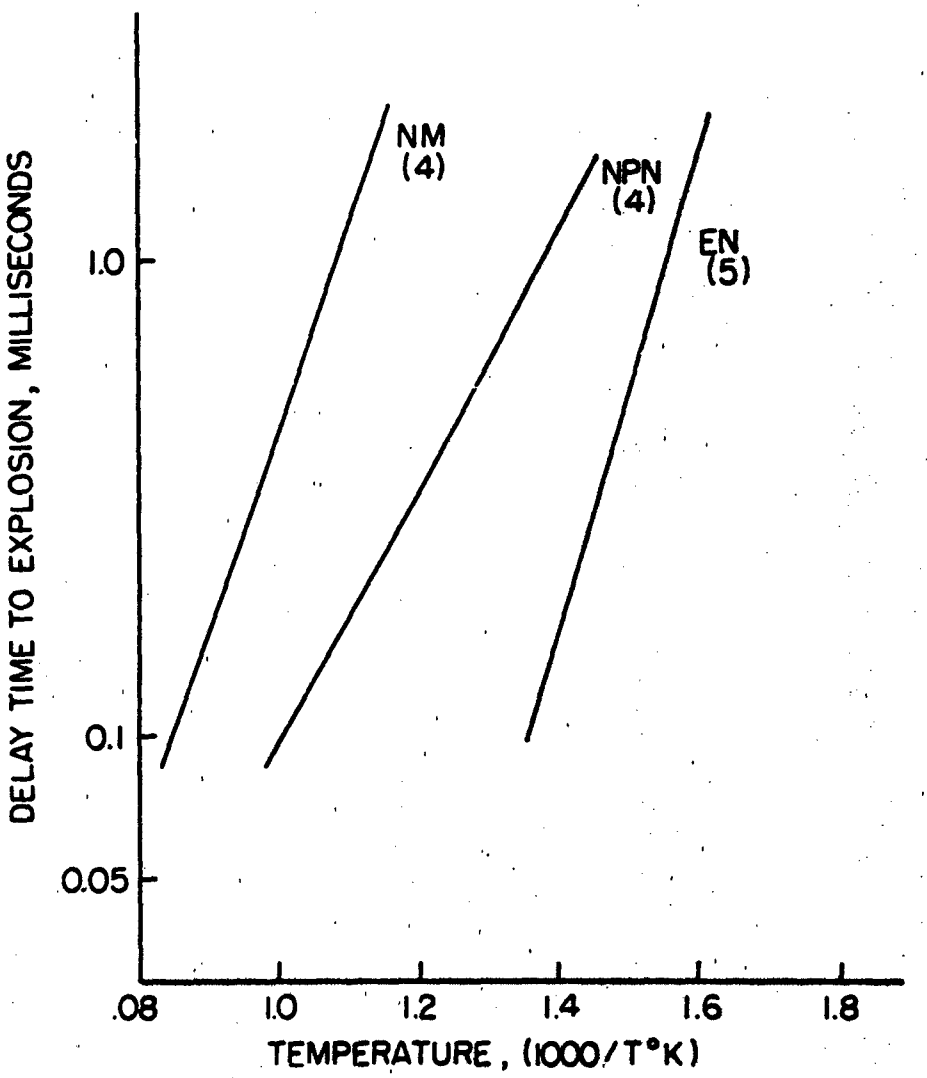


Figure II-1. Typical Thermal Surge Test Results[3,5]

3. P. A. Kendall and J. M. Rosen, "Thermal Initiation Apparatus for High Energy Materials," Review of Scientific Instruments, 39, 7, pp. 992-994, July 1968.
4. J. Wenograd, Transactions Faraday Society, 57, p. 1612, (1961).
5. D. R. Stull, "Fundamentals of Fire and Explosion," AIChE Monograph Series 10, 73 (1977).

5. Thermal Stability. The JANAF thermal stability test is the standard test designed by the ICRPG for testing thermal sensitivity (stability) of propellants. The apparatus consists of a stainless steel cylinder 0.22 inches in diameter by 1.5 inches length closed at one end with a feed-through for a shielded thermocouple. A 0.5 cc (liquid) sample is placed in the cylindrical cavity and the top is sealed with a stainless steel diaphragm 0.003 inches thick. The sample container is placed in a temperature controlled bath which increases at 10°C/minute. The temperature difference between the sample (T_S) and the bath (T_B) is monitored, and temperatures at which thermal activity of the sample (positive $T_S - T_B$ for exothermic reaction and negative for endothermic reaction) are observed are reported.

Isothermal tests are also used to indicate thermal stability. Although there have been several variations on this procedure reported, they are similar in that a sample is placed in a container which is then placed in a controlled temperature bath. The sample temperature (and pressure in some procedures) is monitored for a designated test period, which can be of several days duration. Excursions in temperature or pressure in the sample container are reported as indications of heat of reaction effects.

B. Impact Energy Input Tests

Impact test procedures are designed to simulate rapid compression which may result from mechanical impact directly on the propellant, indirectly on its container, or by adjacent liquid propellant (as in a pumping system). Since compression of gases or vapors results in much higher temperatures than for liquids, most impact test procedures used for evaluation of liquids incorporate gas (or vapor) bubbles in contact with the liquid fuel. The bubble in contact with the fuel is rapidly compressed by means of a free-falling or gas-driven piston. The minimum energy per unit of bubble volume required to initiate observable combustion in the sample is considered a measure of the material's sensitivity to impact initiation.

Thermal explosion theory indicates that impact energy input test results should be dependent on the initial and boundary conditions to which the sample is subjected. Consequently, results from the several impact test procedures, which differ in sample and containment geometry and type of mechanical impetus applied, must be compared with caution.

Two test procedures have been widely used for impact sensitivity testing of energetic liquids. The Drop Weight test recommended by the Interagency Chemical Rocket Propulsion Group (ICRPG) and identified as test No. 4[6] by the Chemical Propulsion Information Agency (CPIA) is now standardized as ASTM D2540-70, Standard Method of Test for Drop Weight

6. *Test No. 4, "Drop Weight Test," Liquid Propellant Test Methods, Liquid Propellant Information Agency, The Johns Hopkins University, Silver Spring, MD (now CPIA, Laurel, MD), December 1959.*

Sensitivity of Liquid Monopropellants. The adiabatic compression test recommended by ICRPG and identified by CPIA as test no. 5[7] is also described by Mead.[8]

1. Drop Weight. A 0.03 mL sample of the liquid is enclosed in a cavity (0.06 mL) formed by a steel cup, an elastic ring, and a steel diaphragm, as shown in Figure II-2. The sample is placed in the steel cup which has an AN 6227B-5 O-ring seated in the bottom. The diaphragm is then placed in the cup so that it drops flat on the O-ring. The cup is then placed in the sample chamber, the piston and ball are fitted, and the top is screwed on with a torque wrench to 7 in-lb. The sample chamber is then mounted in the drop weight assembly which supports a 2-kg drop weight. The weight, which is suspended by an electromagnet, can be released from heights of 0 to 50 cm above the sample container ball. The sensitivity of the material is expressed as the drop height which yields a 50 percent probability of ignition. In the current ASTM test procedure, a test is recorded as positive if the diaphragm is ruptured or if a loud noise or any sign of decomposition such as smoke, charring, gas evolution, or carbon formation is observed.

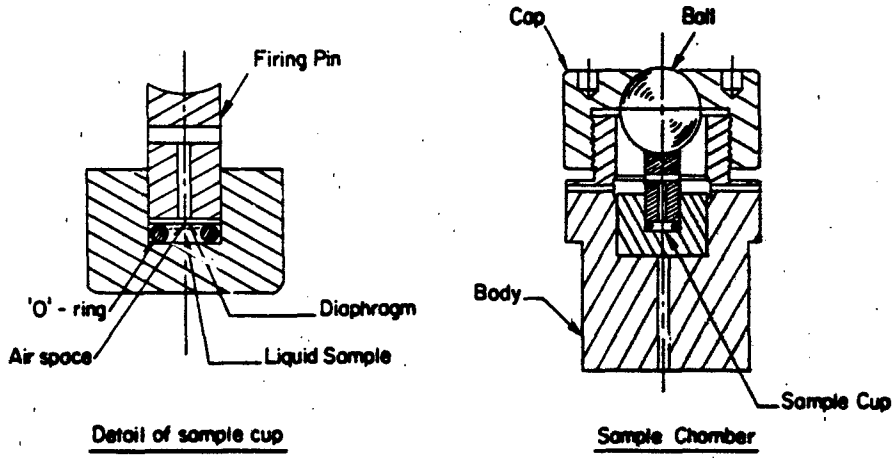


Figure II-2. Drop Weight Test Apparatus

This test requires only a few grams of sample. Test results have been published for a number of energetic materials. The test result is apparatus-dependent and the impact energy required to initiate a sample cannot be simply extrapolated to other test (or actual) configurations. The test result is dependent on the temperature of the sample, as expected from thermal explosion theory. The test configuration dependence is illustrated in one way by the effect of variation of the sample volume (at constant cavity volume) as shown in Figure II-3 for normal propyl nitrate. Smaller liquid sample volumes, at fixed cavity volume, correspond to larger air pockets or bubbles in the test chamber, and it is probable that the

7. *Test No. 5, "Adiabatic Compression Sensitivity Test," Liquid Propellant Test Methods, Liquid Propellant Information Agency, The Johns Hopkins University, Silver Spring, MD (now CPIA, Laurel, MD), December 1959.*
8. *G. A. Mead, "Compression Sensitivity of Monopropellants," ARS Journal, 29, 2, pp. 192-198, 1959.*

results indicated in Figure II-3 are explained by the associated increased heat of air compression for the smaller liquid sample volumes. Typical drop weight test results for several materials, including three solids and four liquids, are shown in Table II-1. Selected drop weight test results published by the Bureau of Mines for several monopropellants are shown in Table II-2, where test results are expressed as the height-mass product which yields a 50 percent probability of ignition.

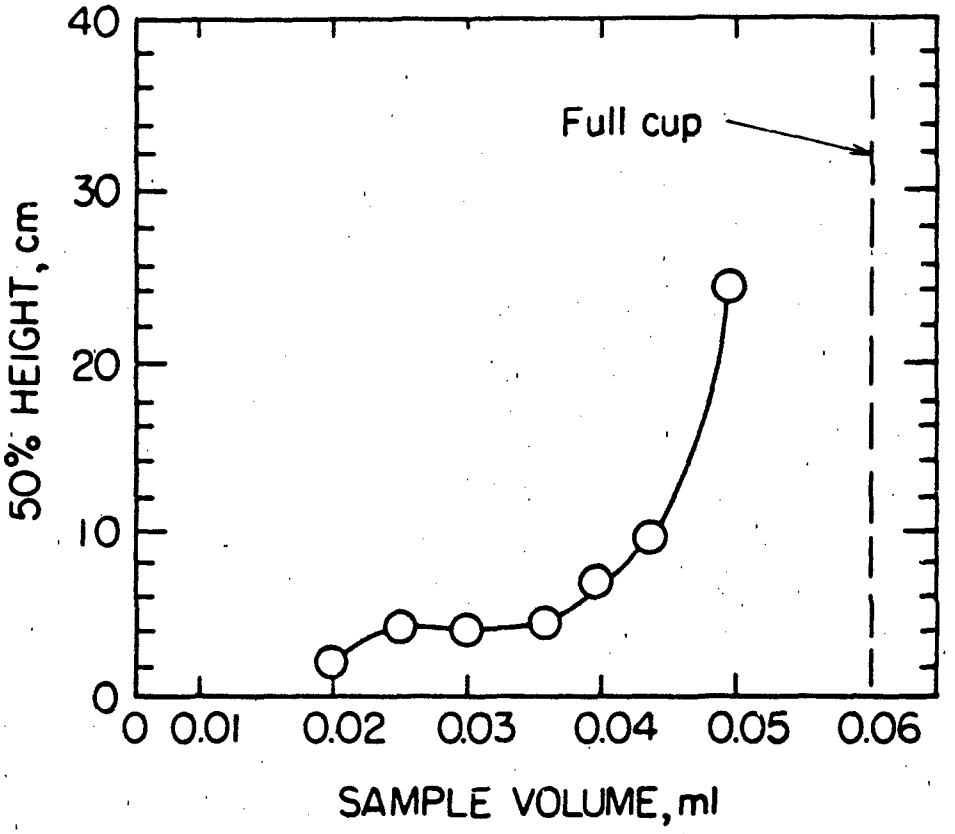


Figure II-3. Sample Volume Dependence-Drop Weight Test of NPN

Table II-1. Typical Drop Weight Test Results (ASTM D 2540)--2 kg Weight @ 70°F

Material	50% Height (cm)
NG (liquid)	1
EN (liquid)	1
NPN (liquid)	4
H (liquid)	>100
HN (solid)	10
RDX (solid)	18
AP (solid)	48

Table II-2. Selected Drop Weight Test Results for Liquid Monopropellants--Bureau of Mines[9,10,11]

Material	Test Condition	Result (kg-cm)	Reference
NPN	70°F, air bubble	17.3 ± 0.58	9
NPN	70°F, CO_2 bubble	167.3 ± 5.3	9
NM	70°F, air bubble	37.3 ± 0.5	9
NM	70°F, CO_2 bubble	163.8 ± 6.94	9
1,2 PDDN	70°F, air bubble	6.92 ± 0.5	9
1,2 PDDN	70°F, CO_2 bubble	113.9 ± 6.1	9
OTTO-I	70°F, air bubble	49.3 ± 2.7	9
OTTO-I	70°F, CO_2 bubble	156.1 ± 4.7	9
OTTO-II	30°C, air bubble, dried	14.7	10
OTTO-II	70°F, air bubble	16.7	11
OTTO-II	70°F, Argon bubble	31.8	11

The variability in results with this test which can be associated with the subjective nature of the criterion for a positive test is also clearly indicated by a series of tests reported by Mason, Ribovich and Weiss[12] on OTTO-II torpedo fuel. Three hundred and fifty tests were made on different samples of the same material, 50 trials each with seven different weights, all dropped from a constant height. Test results were classified according to the following categories:

Post-Test Observation of Sample Container and Piston	Classification
Clean hole in diaphragm	Fast positive
Diaphragm dented or dimpled	Slow positive
Test material remaining, no damage	Negative .

9. *C. M. Mason, J. Ribovich, J. C. Couper and M. D. Weiss,"Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems,"Bureau of Mines Semi-Annual Summary Report No. 3768, July 1, 1959 to December 31, 1959.*
10. *C. M. Mason, J. Ribovich and M. L. Weiss,"Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems," Bureau of Mines Semi-Annual Summary Report No. 3788, January 1, 1960 to June 30, 1960.*
11. *C. M. Mason, J. Ribovich and M. L. Weiss,"Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems," Bureau of Mines Semi-Annual Summary Report No. 3811, July 1, 1960 to December 31, 1960.*
12. *C. M. Mason, J. Ribovich and M. L. Weiss,"Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems," Bureau of Mines Semi-Annual Summary Report No. 3830, January 1, 1961 to June 30, 1961.*

A summary of the results is given in Table II-3.

Table II-3. Drop Weight Test Results for OTTO-II Monopropellant-Variability due to Test Criteria Application[12]

	Energy (kg-cm)						
	10	15	20	25	30	35	40
Number negatives	47	13	1	0	1	1	0
Number fast positives	2	14	23	21	23	23	29
Number slow positives	1	23	26	29	26	26	21

Figure II-4, which is based on the data of Table II-3, gives percent of ignitions observed as a function of weight-height product (kg-cm). The two curves compare the effect of designating dimpled or dented diaphragms (with material reacted) as positive results as opposed to designating only tests where the diaphragm has a clean cut hole as positive. Mason et al. noted that in the range 20 - 40 kg-cm the results appear to be about equally divided between "fast" and "slow" reactions and indicated this behavior had been observed as high as 70 kg-cm. Depending on the interpretation of the test result (note that ASTM D 2540-70 requires a positive result be assigned if any evidence of reaction is observed) a mean value could be assigned for this material from about 20 to about 70 kg-cm. Such results are similar to the problems encountered with the card-gap test (to be described later) when witness plate and container damage are the sole criteria for determining the test result. In view of this observed variability in test results, comparison of drop weight results from different sources should be made with due caution, especially where the exact conditions of test and test criteria are not fully detailed.

2. Adiabatic Compression Sensitivity. A schematic diagram of the test equipment taken from Mead[8] is shown in Figure II-5. The sample consisting of a gas bubble in contact with the liquid is rapidly compressed by a gas-driven piston. Piston velocity (rate of sample pressurization) is varied by adjusting driving gas pressure behind the piston. The sample chamber volume is about 1.3 mL and samples from about 0.4 to 1.1 mL liquid volume and corresponding 0.2 to 0.9 mL bubble volume can be tested. The bubble volume specification is limited by the accuracy of the liquid volume measurement since bubble volume is estimated by difference. The test result is the piston kinetic energy sufficient to cause complete decomposition of the sample (a positive test). As in the drop weight test, the result is dependent on the volume of the gas bubble. Figure II-6 taken from Mead[8] shows the effect of the bubble volume on the piston kinetic energy required for initiation of normal propyl nitrate. Since the resulting curve is linear, the result can be expressed as 6.7 ± 1.2 kg-cm/mL. The points marked by o and x designate the average of negative results and the average of positive results respectively and give some indication of test repeatability. It was noted, however, by Mead that at V = 0.8 mL positive

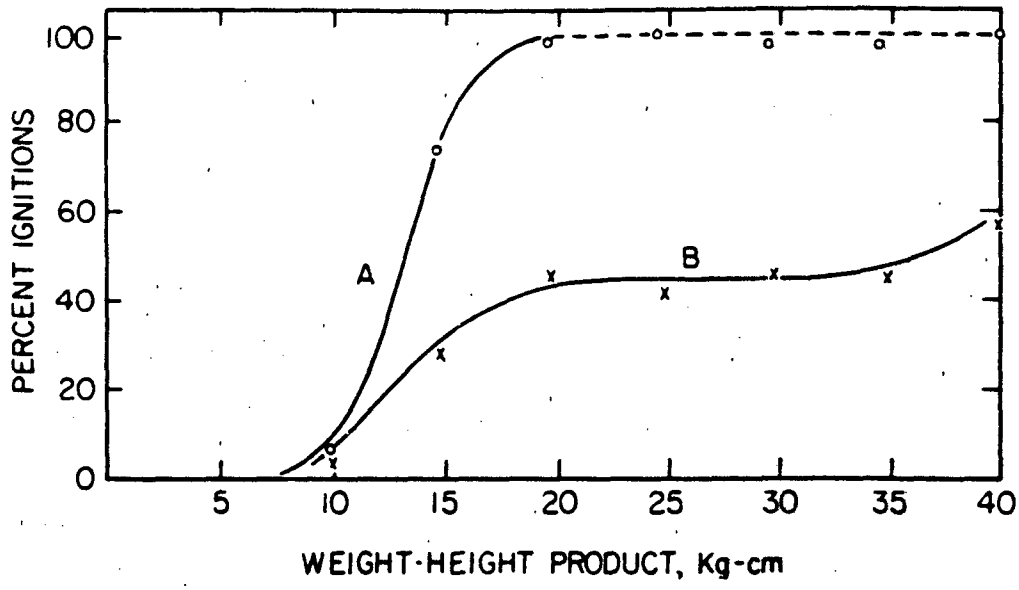


Figure II-4. Effect of Subjective Nature of Test Criteria-Drop Weight Test--Curve A, Any Evidence of Reaction Designated as Positives--Curve B, "Partial" Reactions Designated as Negatives--OTTO-II[12]

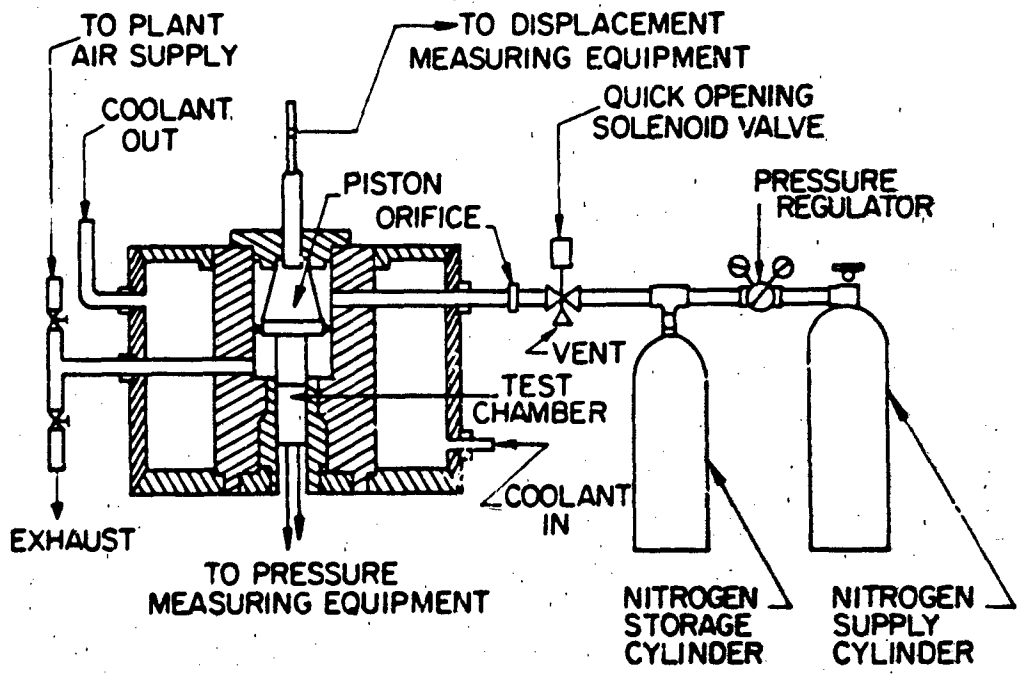


Figure II-5. Schematic Diagram Adiabatic Compression Test[8]

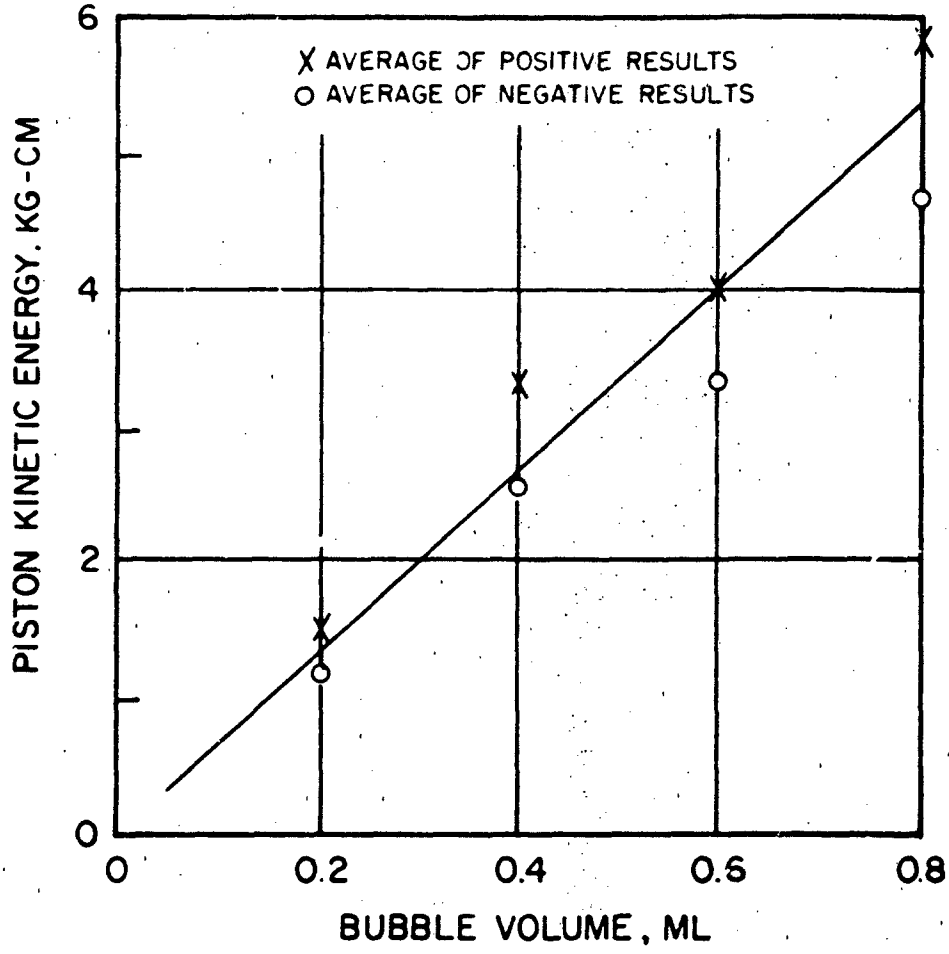


Figure II-6. Bubble Volume Dependence--Adiabatic Compression Test of NPN[8]

results were occasionally obtained for normal propyl nitrate at energy inputs as low as 3.2 kg-cm and negative results as high as 5.9 kg-cm. Results for other materials reported by Mead are given in Table II-4. Table II-5 gives selected results of adiabatic compression sensitivity for normal propyl nitrate and two candidate torpedo fuels as reported by the Bureau of Mines. Some of the results in Table II-5 are based on measurement of piston velocity while others are based on correlations of piston velocity with pressure developed for the apparatus, as in Mead.[8] The Bureau of Mines work cited in Table II-5 indicates that considerable scatter in results can occur because of problems in repeatability of piston velocity from test to test.

3. Compression-Ignition Sensitivity of Liquid Gun Propellants at Gun Operating Conditions. Studies have been conducted to determine the sensitivity of liquid gun propellants to compression energy input under conditions designed to simulate those encountered in gun operations. A description of the methods and procedures used is reported in Part III of this report, which summarizes the LGP safety test data reviewed in this work.

Table II-4. Adiabatic Compression Sensitivity Test Results for Liquids Reported by Mead[8]

Material	Result (kg-cm/ml)
EN/PN 60/40	4.0 ± 0.8
NPN	6.7 ± 1.2
NM	10.4 ± 1.7
Methylacetylene	86 ± 12
Hydrogen peroxide	> 144 (equipment limit)
H	> 144
UDMH	> 144
EO	> 144

Table II-5. Selected Adiabatic Compression Sensitivity Test Results for Liquid Monopropellants--Bureau of Mines[11, 12, 13]

Material	Test Condition	Result (kg-cm/ml)	Reference
NPN	Air bubble	6.6 ± 0.7	11
NPN	Air bubble/small sample	9.5	11
NPN	Air bubble (0.7 ml)	4.6	13
NPN	CO_2 bubble	26.0 ± 2.6	11
NPN	CO_2 bubble	27.5 ± 2.0 - 6.0	11
OTTO-I	Air bubble	14.2 ± 1.4	11
OTTO-I	Air bubble	15.2 ± 1.8 - 1.4	11
OTTO-I	Air bubble	21.7 (dried)	11
OTTO-I	CO_2 bubble	22.7 ± 2.3	11
OTTO-I	CO_2 bubble/small sample	21.0	11
OTTO-II	Air bubble (0.7 ml)	7.6	13
OTTO-II	Air bubble	21.8	12
OTTO-II	Argon bubble	29.1	12

4. Other Low Amplitude Compression Wave Tests. Hay and Watson[14] have described a test to simulate the development of explosive reaction (defined for this test as any chemical reaction releasing gases and energy rapidly enough to cause rupture of the container and displacement of surrounding objects) in a large mass of cavitated liquid. A schematic diagram of the equipment is shown in Figure II-7. The liquid sample is contained

13. C. M. Mason and J. Ribovich, "Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems," Bureau of Mines Semi-Annual Summary Report No. 3897, January 1, 1963 to June 30, 1963.
14. J. E. Hay and R. W. Watson, "Initiation of Detonation in Insensitive Liquid Explosives by Low Amplitude Compression Waves," Sixth Symposium (International) on Detonation, San Diego, CA, August 24-27, 1976.

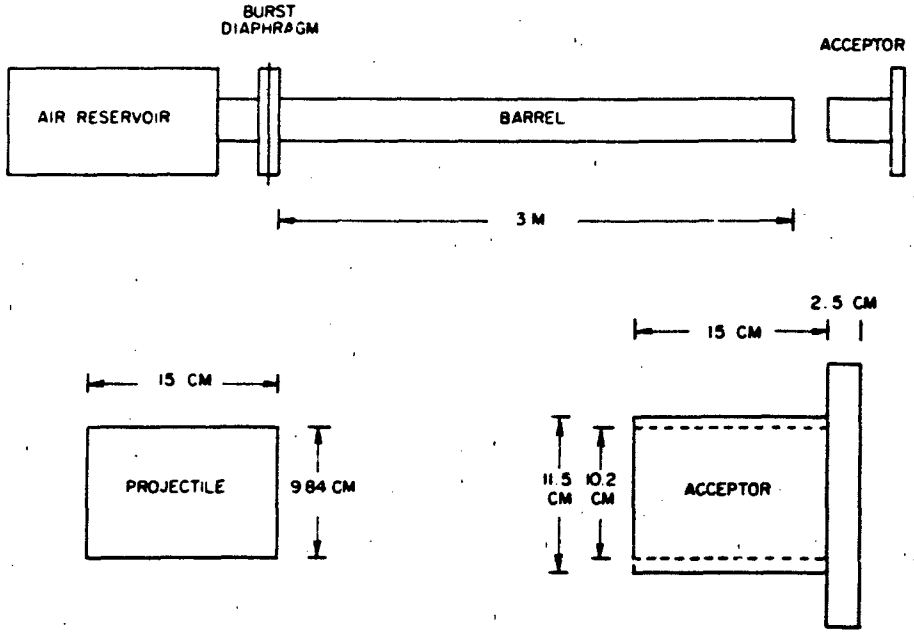


Figure II-7. Low Amplitude Compression Wave Impact Test[14]

in a steel cylinder of 10.2 cm ID, 15 cm length, 1.27 cm wall thickness, with a 2.5 cm thick steel plate welded to one end. The sample is retained in the cylinder by a 0.0076 cm thick polyethylene diaphragm fastened over the open end. Air bubbles are introduced into the liquid by means of a 15 cm length of PVC tubing (0.24 cm OD, 0.04 cm wall thickness), closed at the end, with two rows of pin holes (0.23 cm diameter spaced 0.3 cm apart) along its length. Air is supplied to the bubbler tube at a gage pressure of 0.55 to 1.4 bars, depending on the properties of the liquid, to maintain a bubble field as nearly uniform as possible from one liquid to another. The initiating stimulus is provided by the impact of a steel projectile 9.84 cm diameter, 15 cm long, weighing 9.4 kg. The projectile is propelled through a steel barrel (10.2 cm ID, 3 m long) by compressed air. The threshold piston velocity which causes an explosion of the liquid is the test result. Table II-6 gives results for several liquid materials, many of which are or have been transported in bulk (e.g. 38,000 liter railroad tank cars). The threshold velocities given in the table are the mean between the highest velocity at which no explosion resulted and the lowest at which explosion resulted. The error interval given is the difference between these values. Hay and Watson noted that nitromethane and 88% monomethylamine nitrate which have reportedly detonated due to impact in transportation accidents,[15,16] show low threshold

15. *Interstate Commerce Commission: Exparte 213. Accident Near Mt. Pulaski, Illinois. 305 I.C.C. pp. 81-87, 1959.*
16. *National Transportation Safety Board. Railroad Accident Report, Burlington Northern, Inc., Monomethylamine Nitrate Explosion, Wenatchee, Washington, August 6, 1974. Report No. NTSB-RAR-76-1.*

velocities for explosion in this test, and suggest correlation of explosive behavior for these materials in this test with conditions existing in such documented transportation accident scenarios.

Table II-6. Low Amplitude Compression Wave Test Data[14]

Material	Test Temperature (°F)	Threshold Velocity (m/sec)
NOS-365	104 and 68	26.2 ± 2.7
NPN	68	91.3 ± 1.3
OTTO-II	68	23.4 ± 3.2
NM	68	24.1 ± 2.3
NM/Benzene 70/30	68	> 114
NM/1/NP 52/48	68	90.2 ± 0.6
NM/2/NP 53/47	68	> 117
NM/toluene 70/30	68	> 122
H	68	> 76
MMAN 88%	165	24.3 ± 5.6
MMAN 69%	165	58.9 ± 5.8
EGMN 75%	68	53.7 ± 7.3
EGMN 50%	68	55.3 ± 8.0
EGMN 38%	68	> 113

C. Shock Energy Input Tests

Several test procedures have been developed for determining the sensitivity to initiation of explosion in a liquid material by shock wave energy input. The test methods described here all share the similarity of energy input to the test material from a detonating "donor" explosive.

1. Card Gap. There are several versions of this test, but all are similar in that the shock from a detonating donor charge is attenuated through an inert material (the "card gap") to a strength barely sufficient to initiate detonation in the material being tested. The amount of attenuation required to prevent detonation of the test material is the practical measure of sensitivity. The greater the attenuation required, the greater is the sensitivity of the material to shock initiation to explosion. The card gap test has been extensively studied and a large amount of test data is available for both solid and liquid explosives and propellants. Much of the development work on it in the United States was done at the Naval Ordnance Laboratory[17] and the test is frequently referred to as the NOL Card Gap Test. The test procedure has been refined in some instances to include provision for additional instrumentation to determine detonation velocities and pressures, which were not obtained in the original test. The Bureau of Mines has an instrumented card gap test which has been used to study shock sensitivity of a large number of materials.

The original version of the NOL Card Gap Test is schematically illustrated in Figure II-8. The basic test assembly includes a steel sample container, a plastic card gap of varying thickness (the shock attenuator), a tetryl donor charge (50.5 grams), an electric blasting cap (No. 8) for initiation of the donor, an alignment tube, and a steel witness plate. In this version of the test, the criterion for a "positive" test (evidence of detonation) is a clean penetration of the 3/8-inch steel witness plate. It has been reported that a peak pressure of 95 Kbar is required to penetrate this type steel plate.[18] Therefore, negative results can be obtained even though the test material undergoes low velocity detonation, where peak pressures of the order of 10 Kbar are anticipated.

Figure II-9 shows the test arrangement recommended by the Bureau of Mines.[19] This version of the card gap test incorporates a pressure measurement near the downstream end of the sample and provision for continuous velocity measurement through the sample length. The basic assembly includes a steel witness plate, steel sample container, a plastic card gap of variable thickness, and a tetryl charge. Detailed specifications for the assembly are given in Table II-7. The velocity probe is attached

17. G. D. Edwards and R. K. Rice, "Liquid Monopropellants: Detonation Sensitivity," NAVORD Report 2884, U.S. Naval Ordnance Laboratory, October 1953.
18. M. A. Cook, "The Science of Industrial Explosives," IRECO Chemicals, Inc., Salt Lake City, Utah (1974).
19. C. M. Mason and E. G. Aiken, "Methods for Evaluating Explosives and Hazardous Materials," Bureau of Mines Information Circular 8541, U.S. Department of the Interior, Bureau of Mines (1972).

Table II-7. Specifications for Bureau of Mines Card Gap Test[19]

Acceptor Container	
Material	Steel
Configuration	Cylinder
Length	Variable
Inside Diameter	1.049 in. (1 in. sch 40)
Wall Thickness	0.133 in.
Bottom Closure	0.005 cm polyethylene membrane stretched over end, retained by rubber band
Explosive Shock Producer (Donor)	
Material	Tetryl
Configuration	Cylindrical pellet
Length	2.54 cm (1 in.)
Diameter	4.13 cm (1-5/8 in.)
Density	1.57 ± 0.03 gm/cm^3
Mass	50 gm
Detonator	No. 8 electric detonator
Shock Attenuator (Card Gap)	
Material	Cellulose acetate
Configuration	Disc
Thickness (per disc)	0.025 cm or 1.27 cm
Diameter	4.13 cm (1-5/8 in.)
Test Criteria	
Linear Burning (or Detonation) Velocity Measurement	
Witness Plate	
Material	Steel
Configuration	Disc or square
Thickness	Variable
Diameter	Variable
Witness Plate Standoff	
Material	Cork
Configuration	Disc
Thickness	0.64 cm (0.5 in.)

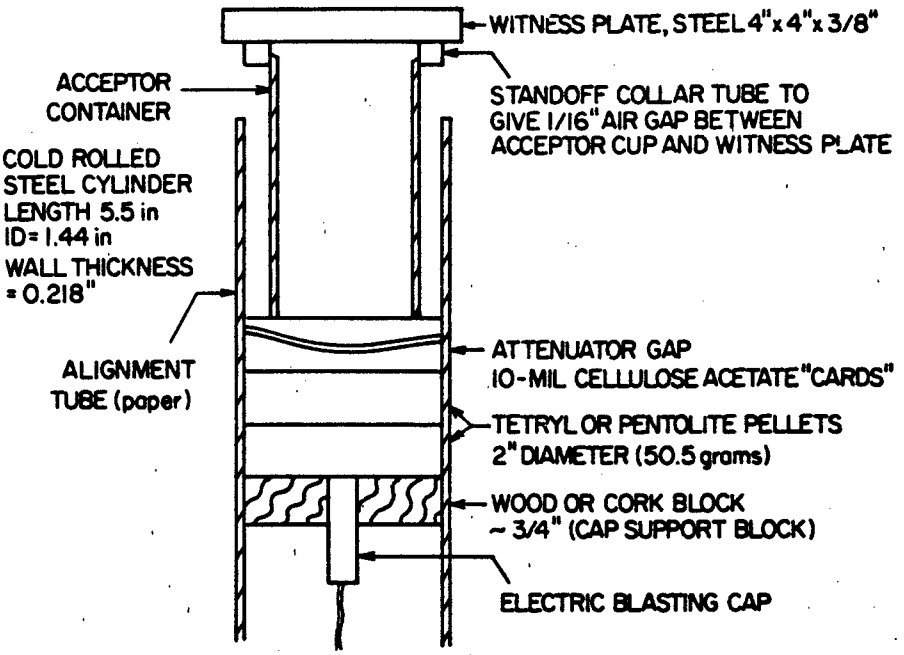


Figure II-8. Naval Ordnance Laboratory Card Gap Test[17]

to the inside or outside wall of the sample container. Details of construction of the velocity probe have been described by Mason and Aiken.[19] Alignment of the assembly is accomplished with a cardboard tube as illustrated in Figure II-9. The result of the test is the attenuator thickness (card gap) that results in detonation of the acceptor 50 percent of the time. Occurrence of detonation is determined from the linear burning (or detonation) velocity, the pressure recorded near the end of the sample, or the damage to the witness plate and container. A summary of card gap tests results obtained with the Bureau of Mines apparatus is given in Table II-8 for a number of propellant and explosive liquid formulations.[20] The acceptor container length was 16 inches. Threshold gap lengths are reported for observation of low velocity as well as high velocity detonations.

2. Impedance Mirror. Mallory[21,22] has suggested the impedance mirror test for measuring induction and reaction times of explosives to determine their intrinsic sensitivity. Figure II-10 shows a diagram of the test equipment, which consists of:

20. R. W. Watson, "Card Gap and Projectile Impact Sensitivity Measurements; A Compilation," U.S. Department of the Interior, Bureau of Mines Information Circular 8605, 1973.
21. H. D. Mallory, "Detonation Reaction Time in Diluted Nitromethane, Journal of Applied Physics," 47, 1, January, 1976.
22. H. D. Mallory, "Detonation Reaction Time in Nitromethane," Physics of Fluids, 19, 9, September, 1976.

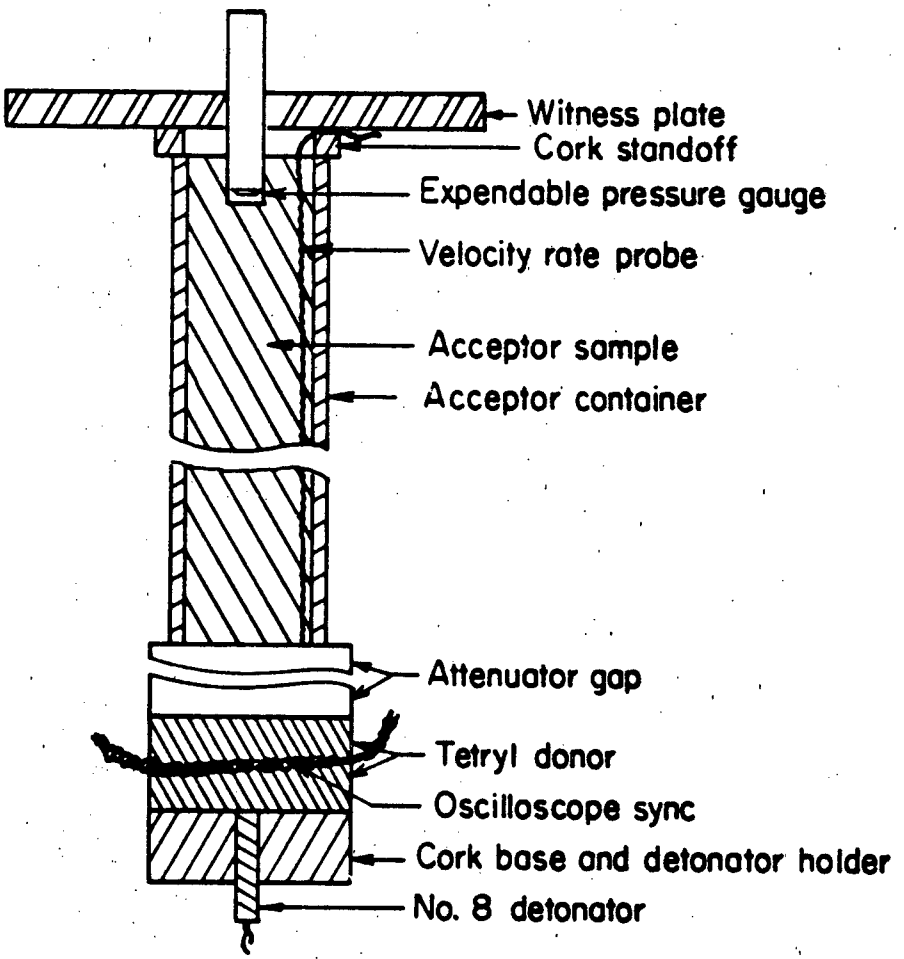


Figure II-9. Bureau of Mines Instrumented Card Gap Test[19]

Table II-8. Selected Bureau of Mines Instrumented Card Gap Test Results[20]

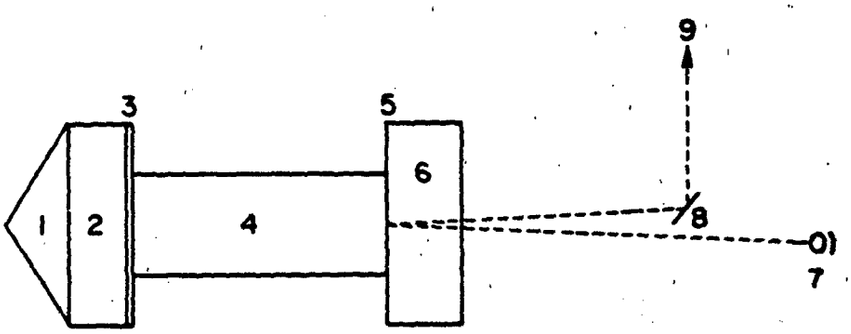
Material		Threshold Gap Length, L, Inches HVD*	LVD**
NG		0.38 < L < 0.5	L > 10.0
NG/EGDN	50/50	0.4 < L < 0.5	L > 10.0
NG/TNM	55.5/44.5	not determined	L > 10.0
NM		0.15 < L < 0.3	none observed
NM/ED		0.5 < L < 0.63	none observed
NM/NG/EGDN	80/10/10	L < 0.5	none observed
	70/15/15	L < 0.5	none observed
	60/20/20	L < 0.5	L > 0.5
NB/WFNA	28/72	10.0 < L < 15.0	none observed
NP/WFNA	32/68	0.05 < L < 1.0	2.0 < L < 5.0
TMETN		0.05 < L < 0.10	L > 10.0
TNM/A	70.5/29.5	L > 10.0	none observed
TNM/BEN	86.3/13.7	L > 10.0	none observed
TNM/OCT	87.7/12.3	L > 10.0	none observed
TEGDN/2-NDDA	99.75/0.25	L < 0.13	none observed
EN		0.10 < L < 0.25	1.0 < L < 2.0
EGDN		L < 1.0	L > 10
H/HN	75/25	No sustained reaction at zero gap	
	70/30	L < 1.0	none observed
	55/45	L < 0.75	L > 0.75
	50/50	0.25 < L < 0.75	5.0 < L < 8.0
	40/60	0.13 < L	L > 10.0
	30/70	0.5 < L < 1.0	L > 10.0
	20/80	0.25 < L < 1.5	L > 10.0
HN/MMH/H	31.4/45.3/23.3	No sustained reaction at zero gap	
	35.0/42.9/22.1	No sustained reaction at zero gap	
	40.0/39.6/20.4	L < 0.5	none observed
	45.0/36.3/18.7	L < 0.5	none observed
	50.0/33.0/17.0	L < 0.5	0.5 < L < 2.0

Table II-8 (continued)

Material		Threshold Gap Length, L, Inches HVD*	LVD**
HN/MMH/N	60.0/26.4/13.6	L < 0.25	8.0 < L < 10.0
	70.0/19.8/10.2	L > 0.13	L < 10.0
	80.0/13.2/6.8	L < 0.25	L > 10.0
	90.0/6.6/3.4	L < 0.13	L > 10.0

*high velocity detonation

**low velocity detonation



Legend:

1. Plane wave lens
2. Composition B disc
3. Aluminum separator plate
4. Explosive tank
5. Saran covered reflective layer
6. Plexiglas mirror substrate
7. Argon-explosive flash
8. Turning mirror
9. To camera

Figure II-10. Impedance Mirror Test Arrangement[21]

a. a plane wave explosive booster composed of a 1 inch thick, 4 inch diameter disk of composition B and a P-40 plane wave lens firing through a 1/8 inch aluminum sheet

b. a test propellant container one end of which is adjacent to the booster and the other end of which is closed with a Plexiglas block with a mirrored surface in contact (through a polyvinylidine sheet) with the test liquid

c. a light beam directed toward the mirrored surface and a turning mirror to direct reflected light to a camera.

When a shock wave from the booster (about 180 Kbar with this arrangement) induces reaction in the liquid propellant in this test setup, the passage of the reactive shock wave at the mirrored surface can be resolved. The arrival and passage of the reactive front is evidenced by fine scale turbulent pressure fluctuations which perturb the mirrored surface, thereby reducing specular reflection from the mirror. With sufficiently thick Plexiglas blocks used as a mirror substrate, the reaction wave can transverse the sample before the Plexiglas block is blown out. The termination of the reactive wave is evidenced by an increase in specular reflection from the mirror. Mallory has reported the reaction time (time for passage of the reaction zone) in steady state detonation of 75/25 nitromethane/acetone to be 0.4 μsec and for pure nitromethane to be 22 ± 3 nsec. Mallory has also tested NOS-365 using the impedance mirror technique. He obtained evidence of detonation (i.e. turbulent pressure fluctuation induced mirror reflectance patterns) using the booster arrangement above. However, the reaction time was very long, of the order of 10 μsec or approximately 500 times that of nitromethane. Furthermore, Mallory found that NOS-365 did not detonate in a charge 58-mm long, 105-mm ID when tested at low temperature (the liquid temperature was not reported, but it was snowing during the test), but did show evidence of detonation at 48-63°C with a propellant charge 305 mm long.

3. Shock-Confinement Tests. Herickes et al.[23] have reported results of tests to evaluate the detonability of systems too insensitive to propagate at zero gap in the standard (NOL) card gap test. The test arrangement referred to as the Confinement Test is shown in Figure II-11. It consists of a 1-inch thick steel target plate as a base, a heavy walled, Type 347 stainless steel tube (2.5 in. OD, 0.5-inch wall thickness, 6.5 inch length) closed at the end with a plastic membrane, and a donor charge. The donor charge is composed of four tetryl pellets (100 grams) and a No. 8 commercial detonator. A Nichrome heating element is provided for heating the sample to the desired temperature before firing. The damage to the tube and the 1-inch thick witness plate gives a qualitative and comparative measure of the sensitivity of the materials tested. Herickes et al. have shown that the sensitivity of nitromethane as measured by the (NOL) card gap test is reduced by the addition of benzene. Approximately 12 percent benzene in NM reduced the card gap value to zero. However, detonation, as evidenced by fragmentation of the tube, was obtained for NM diluted with 20 percent benzene in the confinement test. Figure II-12 shows damage to the steel tube of the confinement test for various benzene-NM mixtures.

23. *J. A. Herickes, J. Ribovich, G. H. Damon and R. W. Van Dolah, "Shock Sensitivity Studies of Liquid Systems," Proceedings of Second Conference on Explosives Sensitivity, Washington, DC, September 16-17, 1957.*

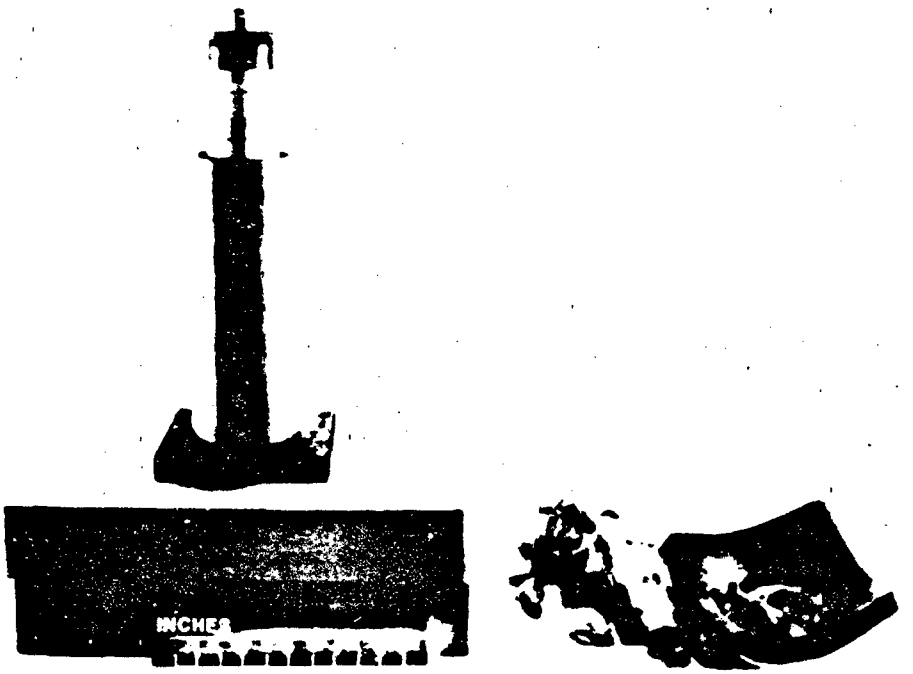


Figure II-11. Bureau of Mines Confinement Test[23]

C_6H_6 (%)	20	30	40	50	100
N.M (%)	80	70	60	50	0

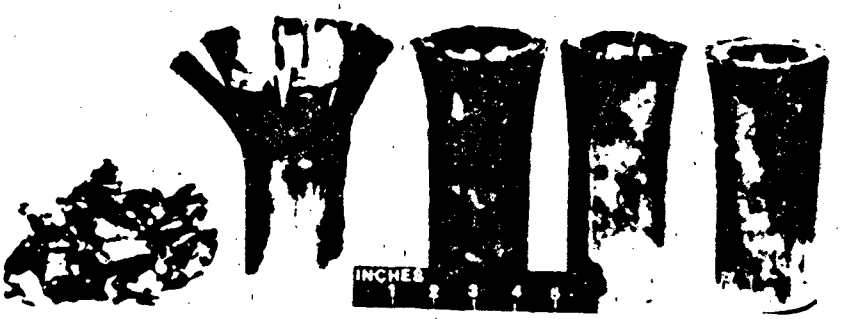


Figure II-12. Confinement Test Results--
Benzene/Nitromethane Mixtures[23]

D. Comparison of Test Parameters and Correlation of Test Results

Sensitivity rating (ordering) of liquid materials is not the same for all the different test procedures which have been described, and comparisons of sensitivity of different materials must be for the same test conditions. Correlation of results from different tests for the same material requires information regarding (1) energy transfer boundary conditions at the test sample surface, (2) conversion of impact or shock wave-deposited energy into thermal energy in the test material, and (3) reaction kinetics as a function of temperature and pressure. At present none of these factors is sufficiently understood to allow quantitative explanation (or correlation) of test results. Furthermore, additional factors such as presence of bubbles of air or vapor from the material, dissolved gases, and surface catalytic effects may be significant in a particular test. However, thermal explosion theory provides a rational basis for at least a qualitative understanding of the effect of many of the test variables. In the following sections, selected information from the literature (primarily from card gap test results) is presented to illustrate the importance of the more important test variables.

1. Initial State of the Sample Material. Thermal explosion theory suggests the initial temperature of a material should affect the result obtained from (for example) the card gap test. Since the ease of attaining a critical (explosion) temperature with a given energy input will depend on the temperature of the material, it is expected that higher card gap values will result from higher test sample temperatures.

Figure II-13 shows the effect of temperature on the card gap test result for OTTO Fuel II as reported by Mason and Ribovich.[24] The test was a modified NOL Card Gap with a 1.05 in. ID, 0.133 in. wall, 3 in. length steel acceptor cup with the standard tetryl charge (50.5 grams). A few points showing the effect of temperature on the card gap value for nitromethane observed by Van Dolah et al.[25] are also included in Figure II-13.

If the fluid material[25] contains a discontinuous gas or vapor phase, deposition of shock energy is expected to result in local hot spots due to (essentially) adiabatic compression of the gas phase. Such a phase might result from dissolved gases (including air) which may be liberated as bubbles due to fluid heating, by mechanical entrainment during fluid transfer operations, or by cavitation. Local temperature increases (hot spots) are expected to be a function of the composition and size of bubbles.

24. C. M. Mason and J. Ribovich, "Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems," U.S. Bureau of Mines Semi-Annual Summary Report No. 3876, July 1, 1962 to December 31, 1962.
25. R. W. Van Dolah, J. Ribovich, J. A. Herickes and G. H. Damon, "Shock Sensitivity of Nitromethane Systems," Communications of XXXI International Congress of Industrial Chemistry, Liege, Belgium, September 7-20, 1958, pp. 121-126.

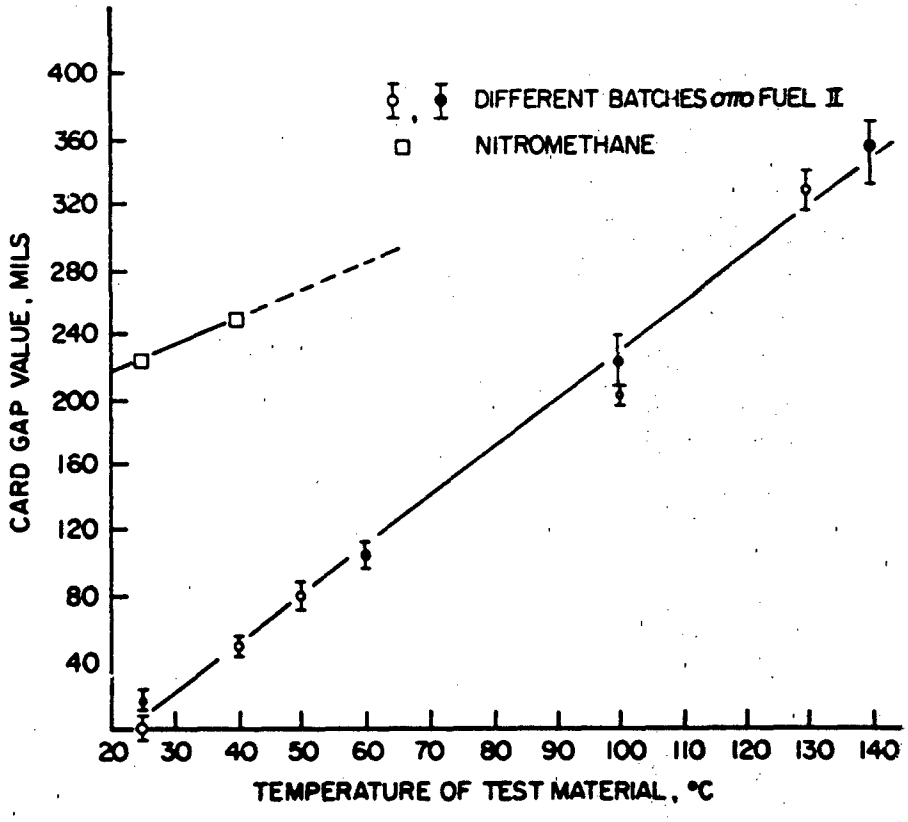


Figure II-13. Effect of Temperature on Card Gap Test Results[24,25]

Figure II-14 shows an experimental arrangement used by Gibson et al.[26] to study the effect of gas bubbles on the shock initiation of low velocity detonation in 50/50 nitroglycerin/ethylene glycol dinitrate (NG/EGDN). High speed photography of these tests appears to demonstrate that chemical reaction is initiated in the immediate area of the bubbles. Simplified calculation techniques have been used by Gibson et al. to estimate the temperature of the compressed bubbles. Estimates of bubble temperatures of approximately 2300°C were made for some of the tests where the material was explosively initiated. Such temperatures would seem to be adequate for initiating chemical reaction, and these analyses strengthen the hypothesis that ignition may occur in such systems at bubble (vapor cavity) sites. From the experiments reported by Gibson et al., it is not possible, however, to conclude that NG/EGDN mixtures containing bubbles are more sensitive than neat mixtures. Although the threshold pressures for initiation to LVD at the donor acceptor interface of NG-EGDN mixtures at 25°C were estimated to be higher in the experiment described in Figure II-14 with gas bubbles than in the standard card gap test without bubbles, the "sensitivities"

26. F. C. Gibson, R. W. Watson, J. E. Hay, C. R. Summers, J. Ribovich and F. H. Scott, "Sensitivity of Propellant Systems," U.S. Bureau of Mines Quarterly Report to Bureau of Naval Weapons for the period January 1, 1966 to March 31, 1966.

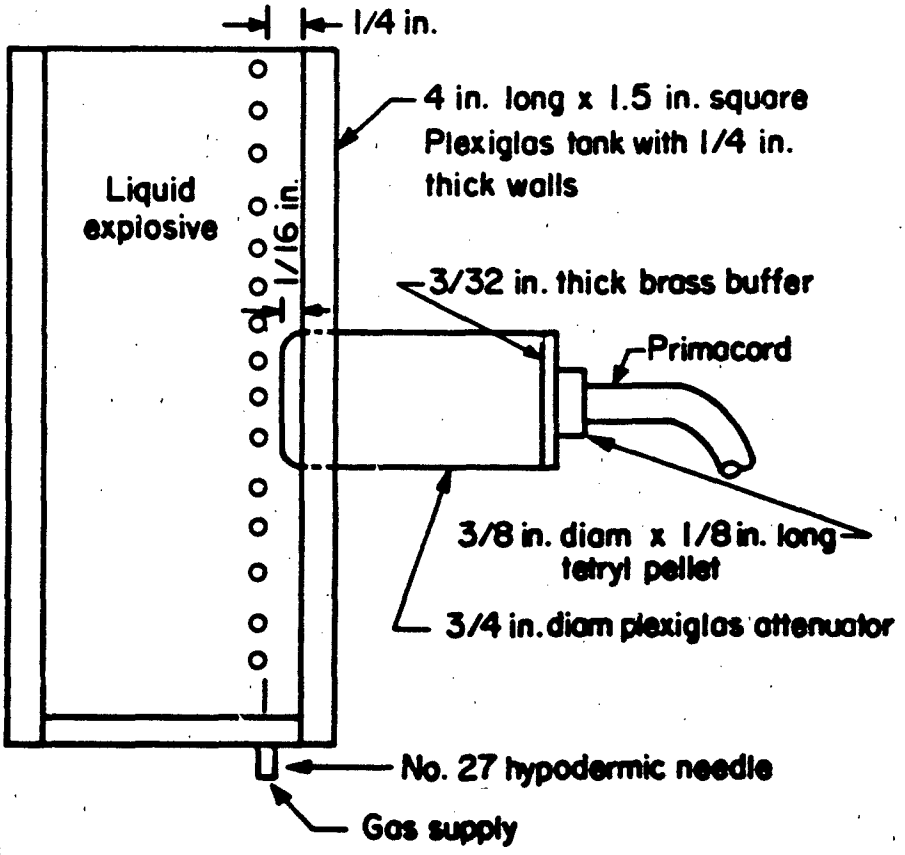


Figure II-14. Apparatus for Studying the Initiation of Liquid Explosives Containing Gas Bubbles[26]

using the two tests cannot be directly compared because of the difference in geometry of the tests. Gibson et al. stated that the apparently lower threshold pressure in the card gap test can be attributed to reflected shock wave interactions associated with the cylindrical geometry of the test.

In any case there appears little doubt that the presence of bubbles of gas or vapor within a liquid propellant or explosive can play an important role in the ignition process. Our understanding of this role in relation to safety testing (and performance testing) is not complete, but there is impressive evidence that the presence of vapor cavities may be directly

associated with establishment of low velocity detonation in explosive liquids and propellants.[27,28]

2. Energy Transfer from Explosive Donor. Since the amount of shock energy deposited in the sample material in the card gap test should increase with increasing explosive donor size, the resulting card gap should increase. Figure II-15 shows the effect of varying donor size on the card gap results for nitromethane presented by Van Dolah et al.[25] The test procedure was a modification of the NOL Card Gap Test, with an acceptor cup of 16ST6 aluminum tubing, 27-mm ID by 76 mm long and 0.89-mm wall thickness. Since increasing donor size in Figure II-15 refers to the use of additional tetryl pellets, the L/D ratio of the donor charge also changes. This geometry effect is probably responsible for the more than doubling of card gap values obtained with doubling of donor amount shown. Van Dolah et al. used a 75-mm square, 25-mm thick steel witness plate. The criterion for evidence of detonation was a dent in the witness plate.

Cook et al.[29] have published calibration curves for the card gap test giving the peak shock pressure at the card gap-water interface as a function of gap thickness. Figure II-16 gives the peak pressure (water) vs. gap thickness for tetryl and pentolite donor systems. Using Figure II-16 and standard impedance matching techniques, the peak pressure entering other test liquids as a function of gap thickness can be estimated. Hence, the card gap test should be expressible in terms of peak shock pressures required to initiate detonation.

3. Boundary Conditions Imposed by Container. As in all other present forms of sensitivity tests, the card gap test identifies susceptibility to detonation under the specific conditions imposed by the test. Extrapolation of card gap test results to the determination of detonation hazard under other conditions of testing or use must be carefully deliberated. Prerequisite to any such extrapolation is some understanding of the effect of boundary containment conditions on initiation and combustion. Card gap test result variability with variations in boundary conditions clearly indicates the need for identification of those parameters which must be considered in hazard evaluation.

Thermal explosion theory indicates there is always an induction time (development time might be a better phrase) associated with the various phases (ignition, deflagration, detonation) of the combustion or explosion

27. R. W. Watson, "The Structure of Low Velocity Detonation Waves," Twelfth Symposium (International) on Combustion, The Combustion Institute, Pittsburgh, Pennsylvania, 1969, p. 723.
28. M. Cowperwaite and D. R. Erlich, "Investigation of Low Velocity Detonation in Liquid Monopropellants and Explosives." Final Report of Contract F44620-73-C-0054 to Air Force Office of Scientific Research, Stanford Research Institute, February 1974.
29. M. A. Cook, R. T. Keyes and W. O. Ursenbach, Journal of Applied Physics, 33, 1962, p. 3413.

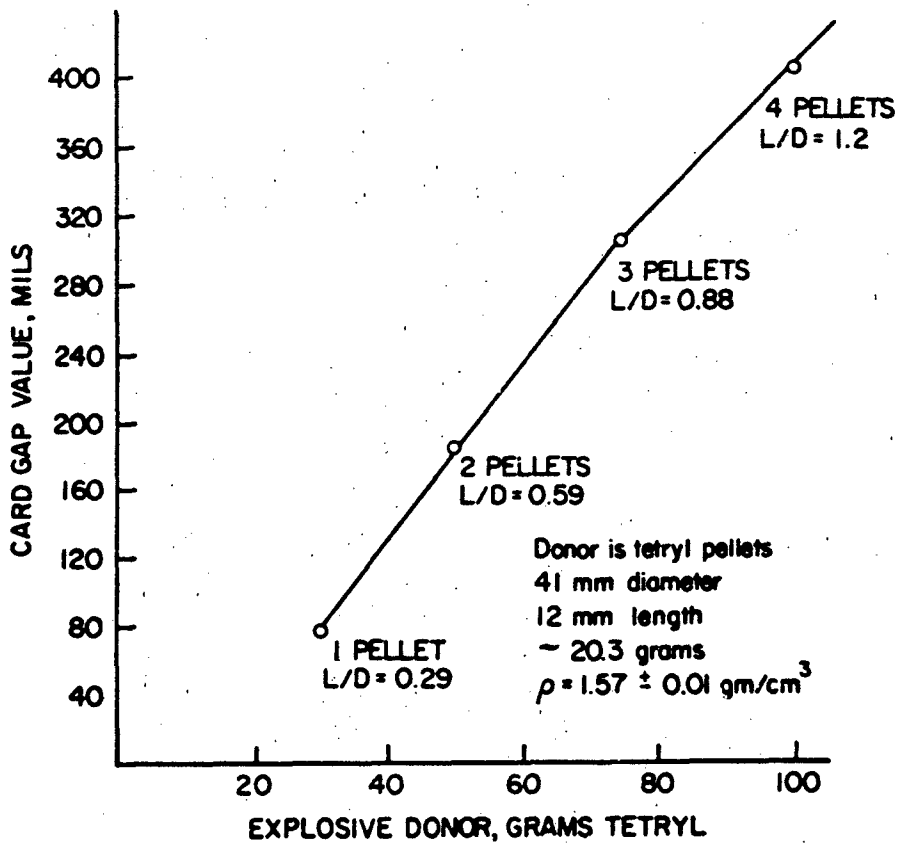
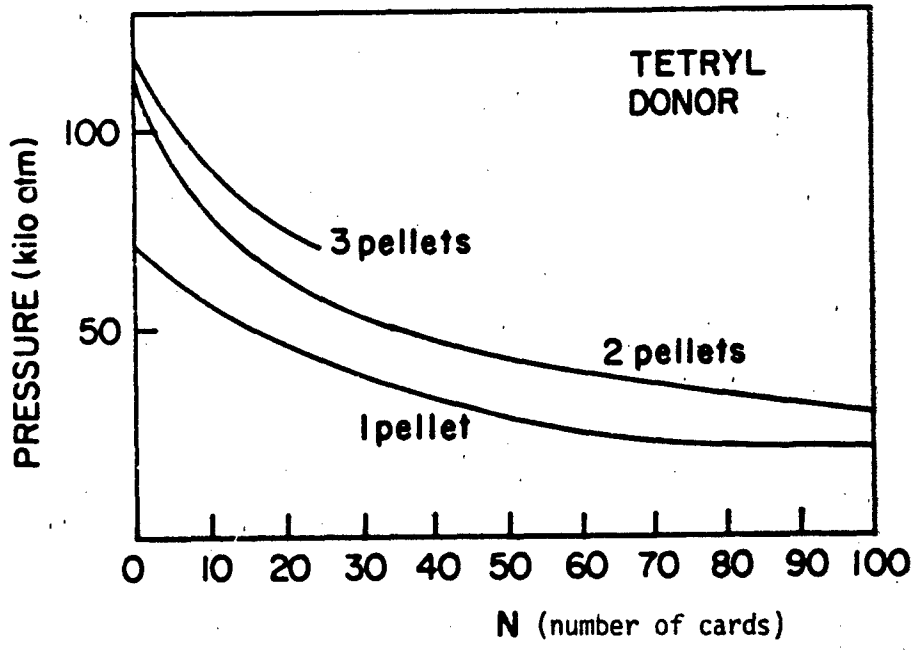


Figure II-15. Effect of Explosive Donor Strength on NOL Card Gap Test for NM[25]

process. Theoretically, all three phases must occur even in a detonating material. In many cases the deflagration phase may be vanishingly short.

The time required for the burning velocity to increase from subsonic values (deflagration) to supersonic values (detonation), indeed whether or not such an increase will occur, depends on the local balance between the rate of thermal energy input and the rate of thermal energy output (transfer). The local rate of energy input can be enhanced by geometrical effects which increase local energy intensity due to shock reflection. The local rates of energy release (transfer away) are affected by the geometry (for example, area for transfer compared with volume for reaction energy deposition) and by the degree of confinement imposed.

In the following sections, selected information from the literature is presented to illustrate the importance of these factors.



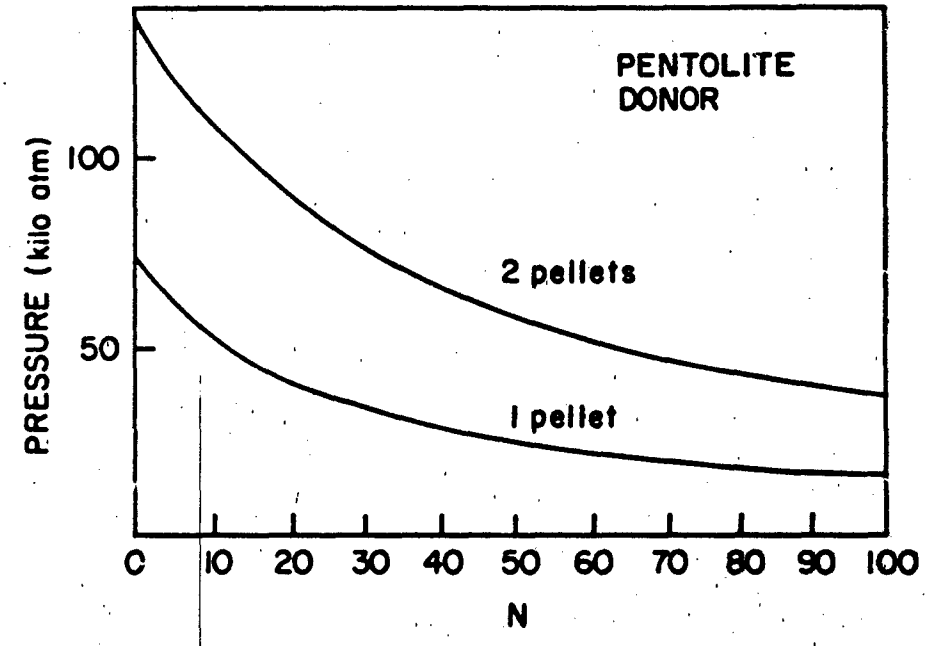


Figure II-16. Peak Pressure vs. Gap Thickness for NOL Card Gap Test[29]

a. Container Geometry. Table II-9 gives card gap test data for 50/50 NG/EGDN at 25°C presented by Mason et al.[10] showing the effect of the geometry of acceptor cup. The Plexiglas cylindrical and square cup results can be compared since the volume of test fluids is the same in both containers. The larger card gap value for the cylindrical cup is probably due to greater shock wave intensity at the center of the cylindrical vessel due to symmetrical reflection of the precursor wave from the acceptor walls. In studies designed to elucidate the mechanism of LVD initiation, Gibson et al.[30] reported that 50/50 NG/EGDN was initiated at 25°C in a cylindrical Plexiglas acceptor cup (1.5 in. ID x 0.125 in. wall x 4 in. long) but not in a square Plexiglas cup (1.5 in. square x 4 in. long) using a 3/8 in. diameter x 3/8 in. long tetryl pellet coupled to a 3/8 in. diameter x 1 in. long Plexiglas rod attenuator. Photographs of this test indicated that the initiation of explosion in the cylindrical sample was associated with localized cavitation along the axis of the sample, presumably from symmetrical precursor wave reflection from the container walls. Although fluid cavitation also occurred in the square tubes, it apparently was not focused along the center of the sample.

Table II-9. Effect of Acceptor Container Geometry on Card Gap* Test Results for 50/50 NG/EGDN at 25°C[10]

Acceptor Container	Card Gap Result (mils)
Plexiglas (cylinder) 0.98" ID x 0.130" wall x 3" length	3140 ± 195
Plexiglas (square) 0.87" ID x 0.130" wall x 3" length	2630 ± 230

*Modified NOL Card Gap Test, 50 grams tetryl, cellulose acetate cards (10 mil thickness) and Plexiglas discs 1/2 or 1 in. thick, target plate 4 in. x 4 in. x 1/4 in., criterion for HVD is sharp hole.

b. Container Material and Thickness. Thermal explosion theory suggests that deflagration to detonation transition should be enhanced by confinement for any system whose burning rate increases monotonically with pressure. It would therefore be expected that a negative card gap result might be obtained for a potentially detonable material due to early container failure, with resultant reaction quenching, before the detonation can develop. Table II-10 gives selected data from the literature on the effect of container material and wall thickness (confinement) on card gap test results. The data are presented in groups in which the only reported variable is the container wall material or wall thickness. The data of Table II-10 clearly demonstrate the variability of card gap test results with

30. F. C. Gibson, R. W. Watson, J. E. Hay, C. R. Summers, J. Ribovich, and F. H. Scott, "Sensitivity of Propellant Systems," Bureau of Mines Quarterly Report to Bureau of Naval Weapons for the Period October 1, 1965 to December 13, 1965.

Table II-10. Effect of Acceptor Container and Wall Thickness (Confinement) on Card Gap Test* Results[10,11,31]

Material	Acceptor Container	Card Gap Result (mils)	Reference
NG/EGDN 50/50	A-1.05-0.035-3	1880 ± 160	31
NG/EGDN 50/50	S-1.05-0.035-3	1500 ± 40	31
NG/EGDN 50/50	S-1.05-0.133-3	515 ± 35	10
NG/EGDN 50/50	P-0.98-0.130-3	3140 ± 195	10
NG/EGDN 50/50	I-1.05-0.133-3	414 ± 157	11
NG/EGDN 50/50	A-1.05-0.133-3	1675 ± 85	11
OTTO-II	A-1.05-0.035-3	0/2 (Positives per no. trials at zero gap)	11
OTTO-II	S-1.05-0.035-3	4/10	11
OTTO-II	GS-1.02/1.33-0.069/0.082-3	0/10	11
NM (99% grade)	S-1.05-0.133-3	~266	31
NM (99% grade)	A-1.05-0.133-3	~256	31

*NOL Configuration, 5.05 g tetryl, 10 mil cellulose acetate cards, 4 x 4 x 1/4" steel witness plate, sharp hole = positive result (HVD)

Container Description 1 - 2 - 3 - 4
1: A - aluminum 61ST6, S = steel, I = iron, P = Plexiglas, GS = glass-lined steel
2: inside diameter, inches
3: wall thickness, inches
4: length, inches

acceptor container material and wall thickness. It is probable that the effects shown can be attributed to the physical mechanisms associated with the container rather than chemical reactivity with the container, although metal surface catalytic effects have been identified, particularly at low card gap values.[18] All of the examples shown in Table II-10 are based on determination of a "positive" test by the presence of a sharp hole in the steel witness plate used, and all are for 3-inch length acceptors. As has been stated previously, overpressures of the order of 95 kilobars are

31. C. M. Mason, J. A. Herickes, J. Ribovich, G. Gelernter and J. C. Couper, "Safety and Combustion Characteristics of Homogeneous and Heterogeneous Monopropellant Systems," Bureau of Mines Semi-Annual Summary Report No. 3748, January 1, 1959 to June 30, 1959.

required to produce this type of response by the witness plate. Hence, a "positive" result is evidence of a high velocity detonation, characterized by velocities and pressures of the order of 6 km/sec and 100 kilobars, respectively.

4. Low Velocity Detonation (LVD). "Low velocity detonation" is characterized by velocities and overpressures of the order of 2 km/sec and 10 kbar respectively, in contrast to "high order detonation" HVD which is characterized by velocities and overpressures of the order of 6 km/sec and 100 kbar respectively. One mechanism for LVD in reactive liquids is associated with fluid cavitation generated by precursor shock waves ahead of the chemical reaction front.[27,28] The resulting cavities serve as reaction centers when compressed by the advancing reaction zone. This mechanism for LVD suggests the reaction is a deflagration induced by a shock wave which provides (through cavitation) sufficient surface area for burning rates capable of supporting the precursor shock. Woolfolk and Amster[32] and Amster et al.[33] have also presented evidence supporting the cavitation mechanism for LVD. They suggested that LVD may also be initiated by shock wave interactions and Mach reflections without the requirement for cavitation. Research is continuing in an attempt to provide models for low velocity detonation behavior.[14,28,34] For hazard evaluation the ability to quantify the potential for LVD is extremely important. The threshold energy inputs which can result in LVD are often much lower than those required for initiation of HVD. It appears that several transportation accidents might be attributed to LVD initiation under circumstances in which shock sensitivity as measured by a standard card gap test (sensitive only to HVD) would not indicate cause for concern.[15,16]

An extensive series of tests has been reported by Mason and Ribovich[35] to determine the threshold gap values (values above which LVD occurred and below which HVD occurred) for 50/50 NG/EGDN in the test arrangement shown in Figure II-17. The test incorporates a 16 in. long sample with provision for timing of shock front passage in the sample using DuPont T_2 target pressure transducers. Figure II-18 shows the threshold gap value separating HVD and LVD initiation in the sample for steel, copper, aluminum, lead, and Lucite (Plexiglas) containers. Note that the gap values in Figure II-18 correspond fairly well with the standard card gap test result for 3 in. long NG/EGDN samples in the same container will thickness combinations shown in Table II-10. However, Figure II-18 indicates that for NG/EGDN the threshold gap value separating the initiation of HVD from the

32. R. Woolfolk and A. Amster, "Low Velocity Detonations: Some Experimental Studies and their Interpretation," Twelfth Symposium (International) on Combustion, The Combustion Institute, Pittsburgh, Pennsylvania, 1969, p. 731.
33. A. Amster, D. McEchern and C. Pressman, Fourth Symposium (International) on Detonation, ACR-126, Office of Naval Research, 1965.
34. R. Chaiken, "On the Mechanism of Low Velocity Detonation in Liquid Explosives," Astronautica Acta, 17, 1972, pp. 575-587.
35. C. M. Mason and J. Ribovich, "Sensitivity Characteristics of Liquid Explosive Systems," U.S. Bureau of Mines Progress Report No. 6, April 1, 1963 to June 30, 1963.

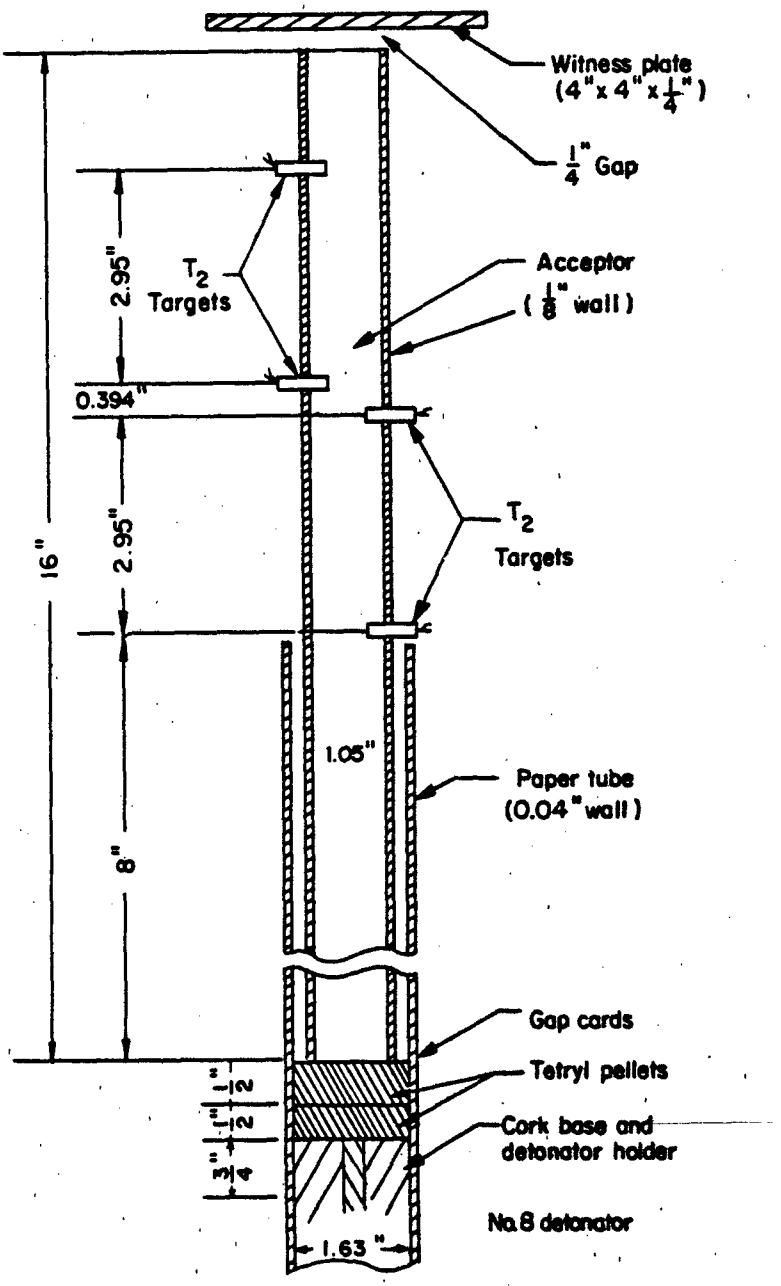


Figure II-17. Bureau of Mines Test Apparatus for LVD/HVD Threshold Determination

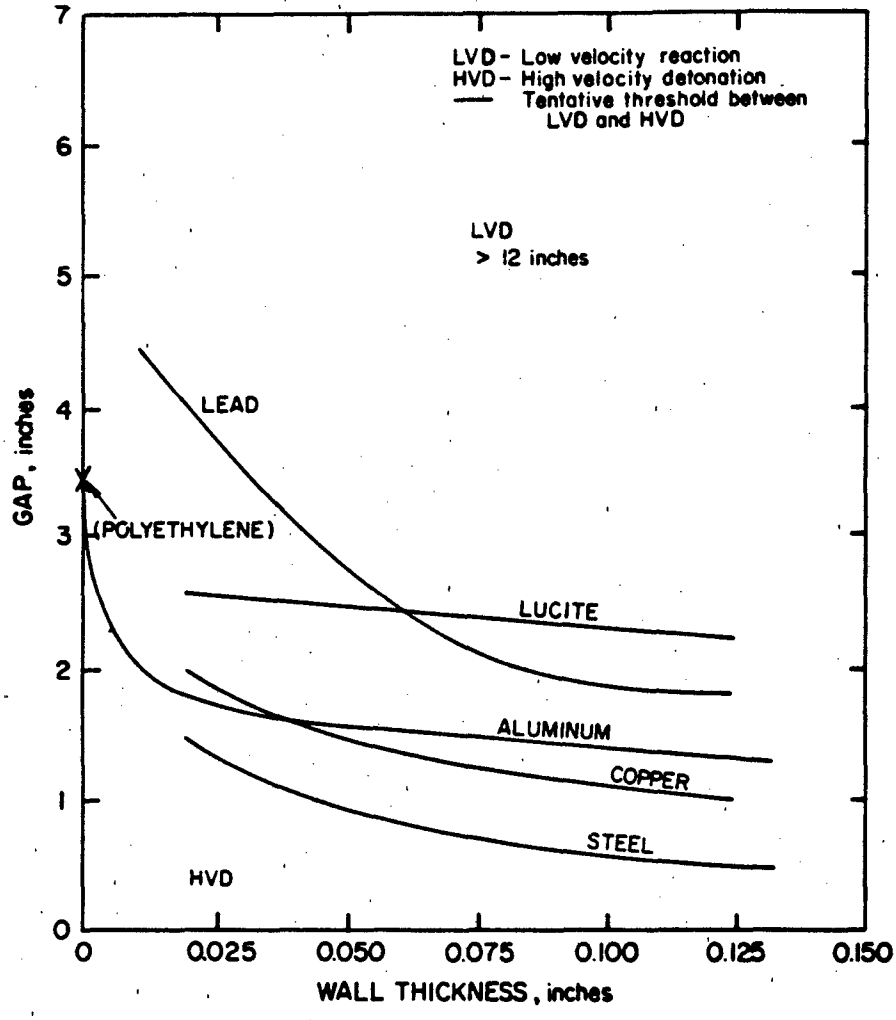


Figure II-18. HVD/LVD Threshold Gap Values for 50/50 NG-EGDN in Different Acceptor Containers[35]

initiation of LVD decreases with an increase in wall thickness. This result contrasts with the usual concept that confinement should enhance the initiation of HVD. However, it can be argued that cavitation might be suppressed in severely confined liquids. Since cavitation has been suggested to be a requirement for LVD and since LVD can under some circumstances transit to HVD, the effect of confinement on cavitation might explain the trend shown in Figure II-18.

III. SUMMARY OF REVIEWED WORK PERTAINING TO SAFETY EVALUATION OF LIQUID GUN PROPELLANTS

This section summarizes test data for liquid gun propellants which were reviewed under this contract. Data reported include information from tests which have been conducted to determine compression sensitivity of specific liquid propellants under simulated gun operating conditions. Although these tests were conducted to determine "safe" gun operating conditions, the data obtained should also be useful in an overall evaluation of risks associated with propellants in storage, handling, and transportation. In some instances, a detailed specification of the test method or procedure was not provided; but in most cases it is believed they correspond to procedures described in Section II, unless specifically noted.

A. Thermal Energy Input Tests

1. Ignition Temperature. Table III-1 gives autoignition temperatures measured by the Setchkin method (ASTM D286-36) for several liquid gun propellant fuels, oxidizers and mixtures.

Table III-1. Autoignition Temperatures[36]

Material	Autoignition Temperature (°C)
2.8 Molar HAN	> 500 (decomposed with white smoke)
11 Molar HAN	> 500 (decomposed with white smoke)
13 Molar HAN	> 500 (decomposed with white smoke)
IPAN	255
TMAN	205
TEAN	410
NOS-365	285
LGP-1776	272
LGP-1845	310

2. Flash Point. Table III-2 gives flash point temperatures, determined by the ASTM 92-72 Cleveland Open Cup Method, for the materials reported in Table III-1.

36. *W. J. Cruice, "Classification of Liquid Gun Propellants and Raw Materials for Transportation and Storage." Contract Report ARBRL-CR-00454 by Hazards Research Corporation, Rockaway, NJ 07866.*

Table III-2. Flash Point (Open Cup) Temperatures[36]

Material	Flash Point (°C)
2.8 M HAN	No flash to boiling temperature (87°C)
11 M HAN	No flash to boiling temperature (87°C)
13 M HAN	No flash to boiling temperature (87°C)
IPAN	No flash to 100°C (liquid at 100°C)
TMAN	No flash to 100°C (still solid)
TEAN	No flash to 100°C (liquid at 100°C)
NOS-365	No flash to 75°C*
LGP-1776	No flash to 75°C*
LGP-1845	No flash to 75°C*

*Not tested at higher temperature due to suspected reactivity hazard condition in this test.

3. Differential Thermal Analysis. Table III-3 gives differential thermal analysis data for several potential liquid gun propellants. Data presented include activation energies (E_A) and frequency factors (k) for decomposition (the sample test atmosphere was not reported) and the onset temperatures (T_{EX}) at which exothermic reactions were observed.

Table III-3. Differential Thermal Analysis Data

Material	E_A (kcal/mole)	k (sec^{-1})	T_{EX} (°C)	Remarks	Reference
OXSOL-1	38.1	0.24×10^{14}	--	DSC*, 40°/min	37
OXSOL-2	42.0	0.78×10^{17}	--	"	37
OTTO-II	17.1	0.78×10^{6}	--	"	37
NOS-365	84.3	0.42×10^{38}	--	"	37
NOS-365	--	--	167/187	DSC, 20°/40° min^{-1}	38
NOS-365	--	--	180	DSC	39
NOS-5	--	--	160	DSC	40

*differential scanning calorimeter

37. B. Smith and J. M. Harrison, "Comparison of Solid and Liquid Gun Propellants," Naval Surface Weapons Center Report NSWC/DL TR-3341.
38. B. Smith, J. Harrison, R. Gibbs, and J. Garrison, "Binary Explosives," Naval Surface Weapons Center Report NSWC/DL TR-214, October 1974.
39. E. S. Romero, "Liquid Propellant Technology", Naval Weapons Center Report NWC-TM-2458, August 1974 (AD-C000-800).
40. Cziesler, Gotzmer, Mueller, Nauflett and Wagaman, "Liquid Aqueous Monopropellants: Part I," Indianhead Technical Report TR 341, July 30, 1971.

4. Thermal Surge. Table III-4 gives thermal surge test data for several liquid propellants. Data are presented as the measured temperature required to give a 250 μsec delay (before explosion) under the test conditions described in Section II.

Table III-4. Thermal Surge Test Data[41]

Material	T (°K) for 250 μsec Explosion Delay
NG	277
NG + 1.5% NDPA	325
DNP	367
1,2 DNG	369
1,2 DNG + 1.5% NDPA	383
1,3 DNG	370
EGMN	518
EGMN + 1.5% NDPA	547
EGMN:H_2O 90/10	666
EGMN:H_2O 80/20	816
1 MNG:1,2 DNG:H_2O	
80/10/10	734
70/20/10	477
60/20/20	797
55/20/25	985
OTTO-II	611

5. Thermal Stability. Table III-5 gives JANAF thermal stability test (see Section II for test description) results for the same liquid gun propellant fuels, oxidizers, and mixtures reported in Table III-1.

In addition to the JANAF thermal stability tests described in Table III-4, Cruice[36] has reported results of two additional thermal stability tests on the materials reported in Table III-1. Table III-6 gives results reported by Cruice for a "long term" thermal stability test. A sample of the material was placed in open or closed glass cups in a stainless steel bomb (net volume = 280 cc) equipped for continuous temperature and pressure monitoring. The bomb was placed in an oil bath and brought to 100°C (or an appropriate lower temperature) and the sample was monitored for 48 hours for temperature and/or pressure excursions. Cruice stated the absolute values of the temperature or pressure excursions are not highly reliable, since the purpose of the test was to identify the excursions rather than to quantify them, but that the magnitudes observed were useful in an assessment of the degree of hazard posed by the reactions discovered.

41. C. Boyars and E. Kayser, "Sensitivity of Torpedo Monopropellants," Naval Ordnance Laboratories Technical Report 70-18.

Table III-5. JANAF Thermal Stability Test Results[36]

Material	Temperature of Major Exotherm Onset (°C)	Remarks
2.8 Molar HAN	202	Sharp, rapid exotherm
11 Molar HAN	165	Sharp, rapid exotherm, burst disc
13 Molar HAN	148	Sharp, rapid exotherm, burst disc
IPAN	185, 220	Two sharp exotherms, burst disc
TMAN	None	Weak, sporadic exotherms
TEAN	195	Gradual, smooth exotherm, burst disc
NOS-365	105	Very sharp, very rapid exotherm, burst disc
LGP-1776	145	Very sharp, very rapid exotherm, burst disc
LGP-1845	135*	Very sharp, very rapid exotherm, burst disc

*In a replicate trial, LGP-1845 remained stable to 167°C, then underwent an extremely rapid and energetic reaction, resembling a detonation.

Table III-6. "Long Term" Thermal Stability Test Results[36]

Material	Sample Mass (g)	Temperature (°C)	Results Observed
2.8 Molar HAN	50	100	Open cup, no reaction in 48 hrs.
11 Molar HAN	50	100	Open cup, no reaction in 48 hrs.
13 Molar HAN	50	100	Open cup, rapid temperature increase at 28.5 hrs, P_{max} = 1950 psig
13 Molar HAN	50	75	Open cup, no reaction in 48 hrs.
IPAN	50	100	Open cup, no reaction in 48 hrs.
TMAN	50	100	Open cup, no reaction in 48 hrs.
TEAN	50	100	Open cup, no reaction in 48 hrs.
NOS-365	50	100	Open cup, severe reaction (possibly detonation) at 6.5 hrs., major damage to facility
NOS-365	10	75	Open cup, sudden decomposition at 9.5 hrs., burst 2000 psig disc
NOS-365	10	75	Closed glass cup, no reaction in 48 hrs.
NOS-365	10	100	Closed glass cup, no reaction in 48 hrs.
LGP-1776	50	100	Open cup, no reaction in 48 hrs.
LGP-1845	50	100	Open cup, sudden decomposition at 18.4 hrs., burst 2000 psig disc
LGP-1845	50	75	Open cup, no reaction in 48 hrs.
LGP-1845	10	100	Closed glass cup, no reaction in 48 hrs.

The results reported in Tables III-5 and III-6 indicate that the fuel-oxidizer mixtures are less stable under thermal exposure than are the fuels or oxidizers alone and indicate (Table III-6) that the reactivity observed is metal catalyzed.

Table III-7 gives results of an additional thermal stability scan test performed by Cruice[36] on LGP-1845 and NOS-365 propellants. The test apparatus was the same stainless steel bomb and oil bath used for the long term thermal stability test, but with a glass thermocouple well and a glass cover over the sample container to prevent contact of the test material with the metal bomb parts, and a programmed (nominal) oil bath temperature increase of 2°C/min. A 10 gram sample was used, and pressure and temperature were recorded continuously.

Table III-7. Thermal Stability Scan Test Results[36]

Time from Test Start @ 20°C (min.)	Temperature (°C)	
	NOS-365	LGP-1845
0	20	20
10	28	32
20	40	45
30	52	62
40	65	75
50	80	90
60	95	102
70	106	113
80	117	123
90	127	132
110	143	138*
120	158**	

* @ 109.7 min., T = 147°C and P = 0 psig; @ 109.8 min, T > 200°C, P > 2000 psig

** @ 119.5 min., P = 0 psig; @ 120 min., P = 80 psig; @ 120.5 min., P > 2000 psig

B. Impact Sensitivity Tests

1. Drop Weight. Table III-8 gives drop weight test data (ICRPG Test No. 4) for several potential liquid gun propellants. In some cases the procedure for testing may not have been as described in Section II. For example, the Picatinny test data may reflect use of weights heavier than 2 kg. Such data (where known) are identified in the table.

Table III-8. Drop Weight Test Data

Material	Weight x Height (kg-cm) for 50% Ignition Probability	Reference
OTTO-II	8.5	37
OTTO-II	16.7	11
OTTO-II	13.2	12
OTTO-II	34.2	41
NG	2.5	41
NG + 1.5% NDPA	2.6	41
DNP	5.7	41
1,2 DNG	5.0	41
1,2 DNG + 1.5% NDPA	5.0	41
1,3 DNG	5.0	41
EGMN	6.8	41
EGMN + 1.5% NDPA	6.6	41
EGMN:H_2O 90/10	27.0	41
EGMN:H_2O 80/20	>80	41
1 MNG:1,2 DNG:H_2O		
80/10/10	>80	41
70/20/10	>80	41
60/20/20	>80	41
55/20/25	>80	41
NM	37.3	9
H	>200 (Picatinny tester)	42
H/HN/H_2O 60/35/5	203	42
EN/PN 60/40	5.8	42
PN	15.5	42
PN	17.3	9
Astrolite	56 (Picatinny tester)	38
Astrolite	55 (Olin-Matheson tester)	38
OXSOL-1	115	37
OXSOL-2	112	37
NOS-283	98.2	38
NOS-365	> 100	38
NOS-365	152	36
LGP-1776	162	36
LGP-1845	152	36
2.8 Molar HAN	178	36
11 Molar HAN	168	36
13 Molar HAN	168	36

42. H. Kirshner and M. Silverstein, "Liquid Monopropellants for Guns: A Review and Recommended Research," (AD-361-631), May 1965.

2. Adiabatic Compression. Table III-9 gives adiabatic compression test data (ICRPG Test No. 5) for several materials considered for liquid gun propellant application.

Table III-9. Adiabatic Compression Test Data

Material	Test Result (kg-cm/ml)	Reference
OTTO-I	14.2 ± 1.4	10
OTTO-II	21.8 (air bubble)	11
OTTO-II	7.6 (air bubble 0.7 ml)	13
OTTO-II	14.5	40
NPN	6.7 ± 1.2	8
NPN	6.6 ± 0.7	10
NPN	4.6	13
EN/PN 60/40	4.0 ± 0.8	42

3. Compression-Ignition Sensitivity Measurements at Gun Operating Conditions. Studies have been conducted at the General Electric Ordnance Systems Laboratories and Princeton Combustion Research Laboratories to define gun operating conditions which will prevent premature ignition of a specific liquid propellant charge due to compression of air or vapor bubbles introduced in the propellant charge filling process. The test procedures were designed to simulate gun operating conditions. However, correlation of results from these tests with those from other impact sensitivity test procedures, as well as shock sensitivity studies which have addressed the ignition mechanism of bubble compression, might provide information on the response of such liquid propellants to a wide spectrum of energy input stimuli, and such information could be used to assess the potential hazards of the propellant under exposure conditions which may be encountered in handling and transportation.

a. General Electric Studies.[43,44,45] Tests were performed to determine the ignition sensitivity of NOS-365 liquid gun propellant to compression of occluded ullage under quiescent conditions and rapid chamber filling conditions (rapid filling conditions may involve cavitation in the liquid which can control the amount and bubble size distribution of the gas phase). The tests were designed to evaluate the effect of the following variables.

43. J. Mandzy, K. Schaefer, J. Knapton and W. Morrison, Progress Report on "Compression Ignition Sensitivity of NOS-365," CPIA Publication 315, Vol. I, March 1980, pp. 377-398 (1980 JANNAF Propulsion Meeting).
44. J. Mandzy, K. Schaefer, J. Knapton and W. Morrison, Progress Report on "Compression Ignition Sensitivity of NOS-365 Under Rapid Propellant Fill Conditions," CPIA Publication 329, Vol. II, November 1980, pp. 309-327 (17th JANNAF Combustion Meeting).
45. W. Morrison, J. Knapton and J. Mandzy, Progress Report on a "Mechanism for the Compressive Ignition of Liquid Monopropellants," CPIA Publication 329, Vol. II, November 1980, pp. 287-307.

Total ullage, U

Peak pressure, P_m

Average rate of pressure increase, $\dot{P}_{avg}$

Maximum sustained rate of pressure increase, $\dot{P}_m$

The maximum sustained rate of pressure increase $\dot{P}_m$ is a measure of the rate of pressurization that occurred during the highly oscillatory sample pressure response observed in some of these tests. The test equipment used differed for the slow and rapid fill conditions, and these will be described separately.

Figure III-1 is a schematic of the test fixture used for the quiescent condition/compression ignition studies. The propellant sample, along with a predetermined amount of ullage, is sealed in a flexible plastic tubing section (the "squeeze tube" in Figure III-1) of nominal 21 cc volume and placed in the test chamber, which is completely filled with water. The water-filled test chamber is terminated at one end with a floating piston which separates the test chamber from a combustion chamber where pressure is generated by burning a solid propellant charge. The pressure pulse shape (determining P_m, $\dot{P}_{avg}$ and $\dot{P}_m$) is adjusted by controlling the amount and burning rate of the solid propellant. Damping of the pressure response of the piston and water reservoir, utilizing water ejection through ports placed in the water reservoir wall, was incorporated to make the pressure applied to the test sample correspond more closely to conditions observed in the propellant reservoir of a liquid propellant gun.

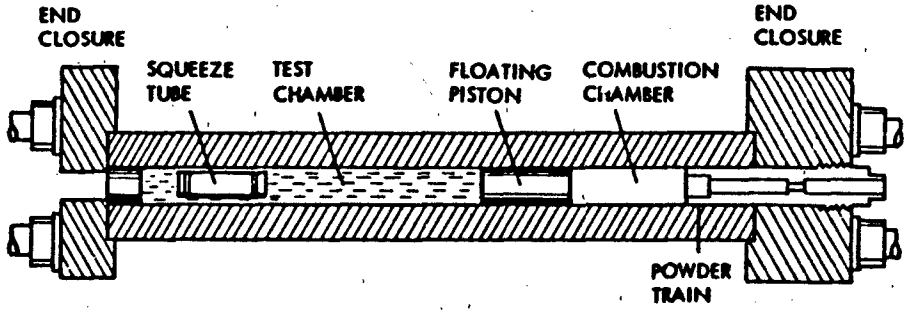


Figure III-1. General Electric Slow Fill Compression Ignition Sensitivity Test Apparatus[43]

Pressure measurements were made at different stations in the test and combustion chambers, using piezoelectric pressure gages. Ignition of samples could be determined only by presence of tube damage since ignition events could not be differentiated in the pressure recordings, presumably because of unloading due to early failure and venting of the test apparatus. Thirty-three tests were reported, covering the following ranges of the test parameters:

$0 < \text{ullage} < 2.0\ cm^3$.

Peak pressure $\leq$ 120 kpsi

Average pressure increase rate $\leq$ 413 kpsi/msec

Maximum sustained pressure increase rate $\leq$ 3400 kpsi/msec

In the thirty-three tests reported, two definite and four "possible" ignitions were observed. For ullages up to 2.0 cm^3, no ignitions were observed in the test parameter space: $P_m < 55$ kpsi, $P_{avg} < 80$ kpsi/msec, $\dot{P}_m < 1000$ kpsi/msec. It was noted that the ignition events observed appeared uniformly distributed with respect to the variable U, but it was observed that the ullage (in samples removed intact, i.e. no ignition or tube failure) had been broken into many smaller bubbles with diameters of the order 1 mm. It was therefore hypothesized that the bubble size which was determining the sample response may have been similar in all tests and independent of the total ullage. It was suggested that ignition was correlated with $\dot{P}_m$, which was determined as the rate of pressure increase measured in the first cycle of the oscillatory sample pressure response, and that the data from these tests could be correlated with the product of the peak pressure P_m and pressure rise rate $\dot{P}_m$.

Figure III-2 is a schematic of the test fixture used in the General Electric rapid filling/compression ignition studies. The test method is similar to the tests under quiescent conditions in that compression is effected by the expansion of gases from a solid propellant charge and controlled by modification of the amount and/or burning characteristics of that charge. A piston terminates the gaseous product (combustion) chamber and is followed by a water volume which, through the use of exit ports in the fixture walls, provides damping of pressure oscillations to provide pressure histories similar to those expected under gun operating conditions. The water damping volume is terminated by a regenerative piston behind which the propellant test charge (22-55 cc) is introduced. The propellant is loaded by a pneumatically driven system using pressurized nitrogen. The ullage to be entrained into the propellant charge is prepositioned in the fill line just outside the test fixture. The filling procedure is designed to minimize cavitation during the filling process and to distribute the ullage in the test chamber (breakup into a fine bubble field). Flow visualization experiments indicated a distributed bubble field with bubble diameter of about 0.25 mm. Nineteen rapid filling tests were reported, covering a range of (liquid) sample volumes from 22 cc to 55 cc and ullage fractions of 0% to 1%. Prepressurization of the samples was 1 kpsi (determined by the pressurized N_2 filling procedure). In nineteen tests two ignitions were observed. It was stated that the pressure rises resulting from these ignitions were "greatly delayed" and would not be observed in gun firing conditions because of the limited time of confinement. Although some pressure records from selected tests were presented, no systematic presentation of maximum pressures or pressure rise rates were reported.

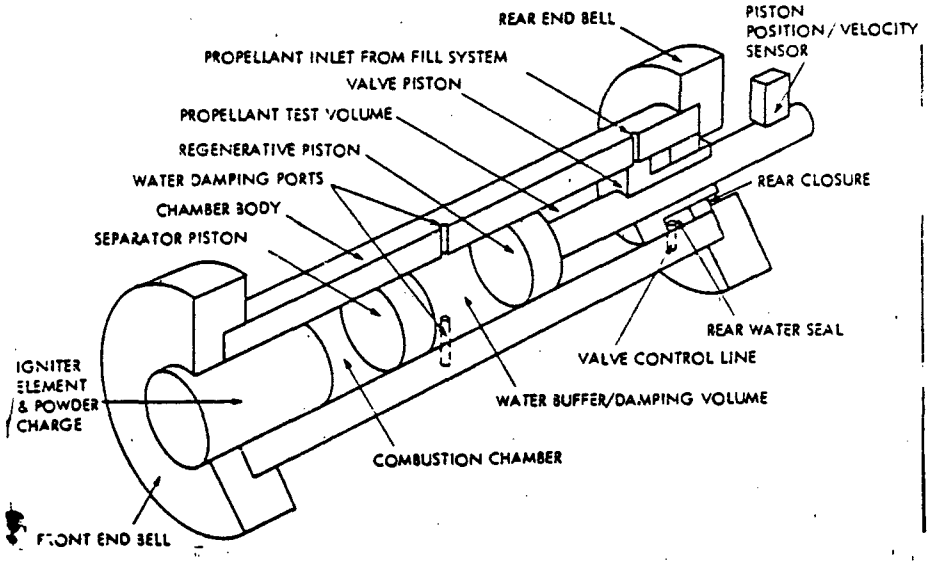


Figure III-2. General Electric Rapid Fill Compression-Ignition Sensitivity Apparatus[44]

b. Princeton Combustion Research Studies.[46,47,48] Tests were performed to determine the ignition sensitivity of NOS-365, LGP-1845 and LGP-1846 liquid gun propellants to rapid compression with different rates of pressure increase and amounts of finely distributed ullage, with and without prepressurization of the propellant charge. Test conditions were designed to encompass those conditions which the propellant would be exposed to in gun operating conditions and to evaluate the effect of the following variables.

Total ullage
Maximum rate of pressure increase
Prepressurization of the liquid charge
(before the onset of the rapid pressure increase)

Figure III-3 is a schematic of the test apparatus used. The propellant is loaded into the pneumatic load cylinder from the LP reservoir. The required amount of ullage is then added to the pneumatic load cylinder

46. *CPIA Publication 347, Volume III, p. 269-287 (1981 JANNAF Combustion Meeting)*

47. *N. Messina, Princeton Combustion Research Laboratories letter report to U.S. Army Ballistic Research Laboratory on Contract No. DAAK11-82-C-0011, Reporting Period January 1, 1982 to March 31, 1982.*

48. *N. Messina, Princeton Combustion Research Laboratories letter report to U.S. Army Ballistic Research Laboratory on Contract No. DAAK11-82-C-0011, Reporting Period April 1, 1982-May 31, 1982.*

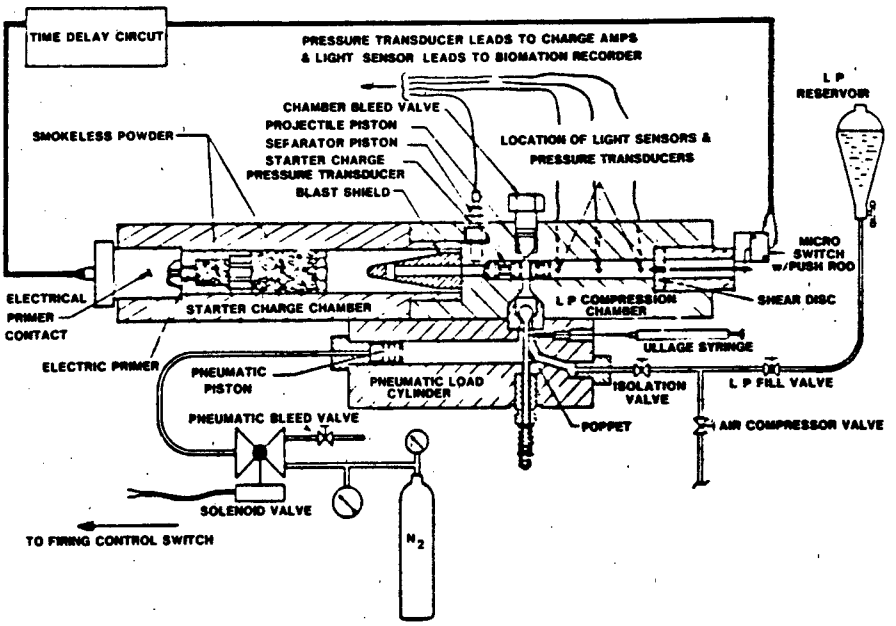


Figure III-3. Princeton Combustion Laboratories[46] Compression-Ignition Sensitivity Apparatus

from the ullage syringe. Rapid filling of the propellant sample compression chamber is accomplished by nitrogen pressurization of the load cylinder. The propellant flows through the poppet valve, which is adjustably spring loaded, into the compression chamber. The spring setting on the poppet valve determines the prepressurization of the propellant charge in the compression chamber. When the propellant charge drives the projectile piston to the end of the compression chamber (the chamber is filled), the piston actuates a time delay circuit which fires an electrical primer actuating the smokeless powder charge in the starter charge chamber. Variable time delays, up to 10 msec, were used to allow for bubble aggregation following the rapid filling process. Pressure buildup due to the expanding combustion gases from the burning smokeless powder drives the separator piston to the right, compressing the propellant charge.

The volume of the sample test chamber is 6.65 cm^3. The pressure pulse shape is determined by varying the type of smokeless powder charge. Maximum liquid pressurization rates (dP_i/dt) of 25, 40, and 70 kpsi/msec were obtained with different types of fuse section and main (combustion) chamber powder charges.

Figure III-4 indicates the results of eleven tests of NOS-365 propellant at different values of the test parameters.

Ullage: 0% (neat) and 3.1%
(bubbles diameter $\bar{d}$ < 0.025 mm)

Injection pressure: 300 and 500 psia

Liquid pressurization rate (kpsi/msec): 25-70

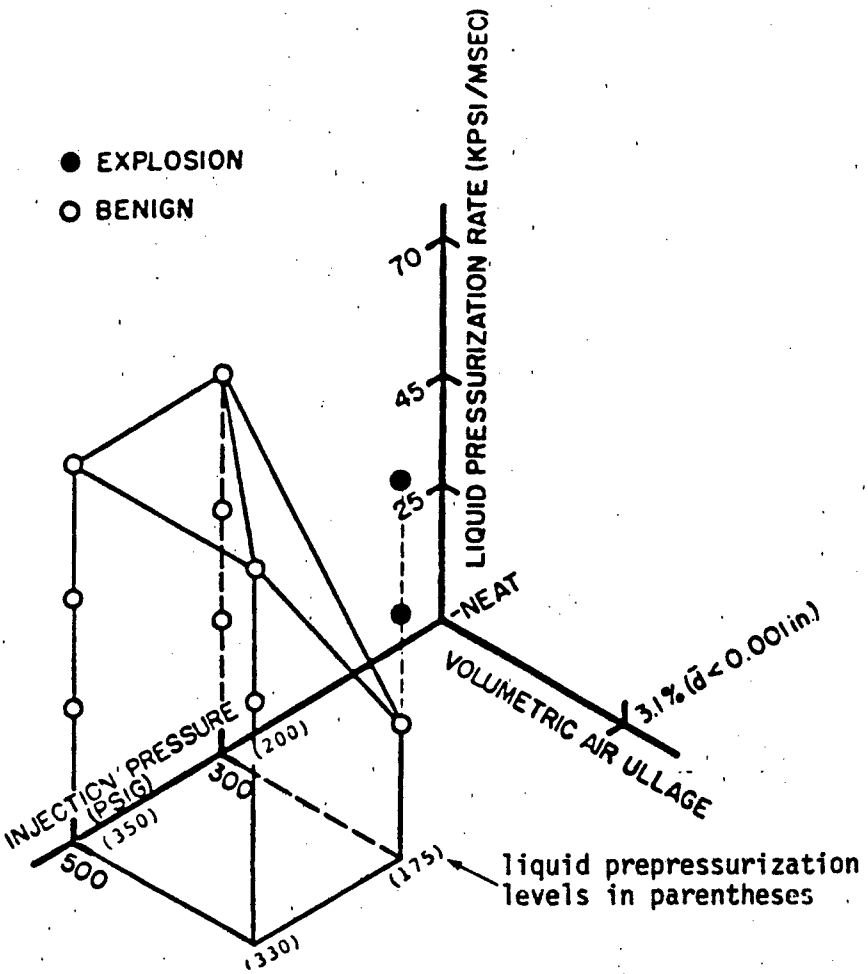


Figure III-4. Domain of Safe Operation for Avoidance of Runaway Reaction due to Compression-Ignition of NOS-365 (from Princeton Combustion Research Laboratories[46])

Figure III-5 shows the results of eleven tests of LGP-1845 for the same test parameters.

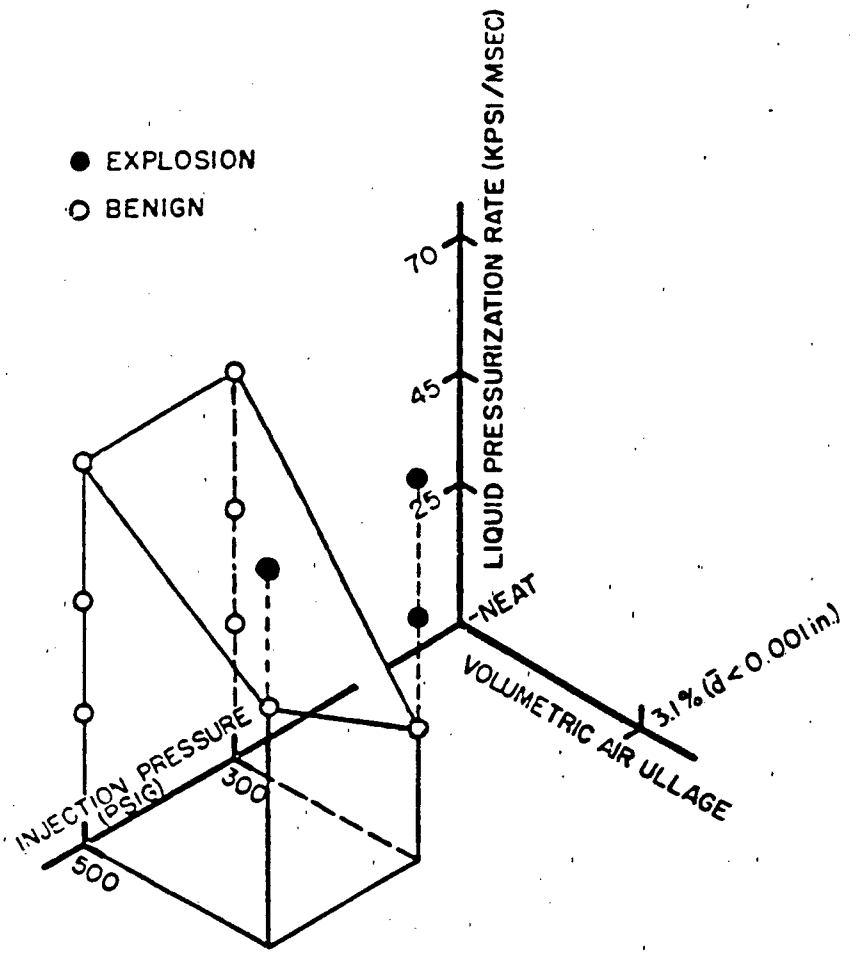


Figure III-5. Domain of Safe Operation for Avoidance of Runaway Reaction Due to Compression-Ignition of LGP-1845 (from Princeton Combustion Laboratories[47])

4. Other Low-Amplitude Compression Wave Tests. Table III-10 (reproduced from Section II) gives the "threshold piston velocity" for explosion of several liquid propellants along with other energetic liquids tested in the low amplitude compression wave test reported by Hay and Watson[14] and described in Section II of this report.

Table III-10. Low Amplitude Compression Wave Test Data[14]

Material		Test Temperature (°F)	Threshold Velocity (m/sec)
NOS-365		104 and 108	26.2 ± 2.7
NPN		68	91.3 ± 1.3
OTTO-II		68	23.4 ± 3.2
NM		68	24.1 ± 2.3
NM/Benzene	70/30	68	>114
NM/1/NP	52/48	68	90.2 ± 0.6
NM/2/NP	53/47	68	>117
NM/Toluene	70/30	68	>122
H		68	> 76
MMAN 88%		165	24.3 ± 5.6
MMAN 69%		165	58.9 ± 5.8
EGMN 75%		68	53.7 ± 7.3
EGMN 50%		68	55.3 ± 8.0
EGMN 38%		68	>113

C. Shock Sensitivity Tests

1. Card Gap. Table III-11 summarizes several card gap test results for liquid gun propellants, and some other energetic liquid materials for comparison, reviewed in this work. In all cases reported in Table III-11 there was at least a strong suggestion that the test was the standard NOL test described in Section II, but details were not given. It cannot be ascertained without a followup contact with the report originators whether the data are all on a comparable test (i.e. same container, etc.) basis. It is noted, for example, that two widely different values for OTTO-II are reported. Where temperature was not specified, it is assumed to have been room temperature or 25°C.

Table III-12 gives "modified card gap test" results for NOS-365, OTTO-II, and hydrogen-hydrogen nitrate-water mixtures reported by Pulsepower Systems, Inc.[49] The test method was reported to be the NOL test with modifications as follows:

49. *Pulsepower Systems, Inc., "Study of Ignition and Combustion of Liquid Propellants for Guns," Monthly Progress Reports TR-140 (Oct. 1977), TR-141 (Nov. 1977), TR-142 (Dec. 1977), and TR-143 (Jan. 1978) to U. S. Army Ballistic Research Laboratory, Contract N00123-73-C-1982, Mod P00013.*

Table III-11. Card Gap Test Data for Potential Liquid Gun Propellants

Material	Card Gap	Remarks	Reference
OXSOL-I	0 (3 tests)	NOL test	37
OXSOL-2	0 (3 tests)	NOL test	37
NOS-5	0		40
NOS-283	0 (3 tests)	NOL test	38
NOS-365	0 (3 tests)	NOL test	38
OTTO-II	120-150 MILS	NOL test	38
OTTO-II	10 MILS		40
NM	190 MILS	NOL test, 0°C	42
NM	470 MILS	NOL test, 80°C	42
H	Insensitive	NOL test	42
EN:PN 60/40	100 MILS	NOL test	42
EO	0 MILS	NOL test	42
EN	200 MILS	NOL test	42
NM	230 MILS	NOL test	42*
NEN	480-500 MILS	NOL test	42

*attributed to Naval Ordnance Laboratory

a. Acceptor containers were 1-7/8 inch OD x 1-7/16 inch ID seamless mechanical (steel) tubing of varying lengths (6-7/8 to 20-7/8 inches).

b. Two 2-inch OD x 1-inch thick pentolite pellets initiated with a No. 8 cap were used as the donor.

c. DuPont T-2 gages were incorporated in the tube wall for measurement of station-to-station average shock velocity.

d. In some tests witness rings were incorporated around the tube to aid in assessing energy release.

e. Provision was made for addition of air bubbles and testing at elevated fluid temperatures.

The last three entries in Table III-12 are for inert liquids to test the effect of shock coupling from the pentolite donor to the liquid in the test container. The water-zinc chloride-ethylene glycol simulated the density and viscosity of NOS-365.

Table III-12. Pulsepower Systems, Inc. Card Gap Test Summary[49]
(all tests at zero card gap)

Material	Tube Length (in.)	Temperature (°C)	Bubbles*	Velocity**, km/sec (Station 1 to Station 2)/ (Station 2 to Station 3)
NOS-365	14	Ambient	No	2.3
NOS-365	20-5/8	Ambient	No	2.1/1.9
NOS-365	20-5/8	Ambient	No	2.2/1.9
NOS-365	20-5/8	Ambient	Yes	2.1/1.6
NOS-365	20-5/8	Ambient	Yes	2.1/1.6
NOS-365	20-5/8	60	Yes	2.1/1.6
NOS-365	20-5/8	60	Yes	2.1/1.6
NOS-365***	20-5/8	60	Yes	2.3/1.8
NOS-365***	20-5/8	60	Yes	2.0/1.6
OTTO-II	13-3/4	Ambient	No	3.8
OTTO-II	20-5/8	Ambient	No	--
OTTO-II	20-5/8	Ambient	Yes	2.9/1.3
OTTO-II	20-5/8	60	Yes	5.7
H-HN-H_2O (63/32/5)	6-7/8	Ambient	No	5.7
H-HN-H_2O (63/32/5)	20-5/8	Ambient	No	9.5/8.2/8.2/6.7****
H_2O	20-5/8	Ambient	No	1.8/1.5
Glycerin	20-5/8	Ambient	No	2.9/1.5
H_2O-ZnCl-EG	20-5/8	Ambient	No	1.9/1.5

*Bubble size reported ~ 1 mm diameter.

**Station distance from tube bottom: #1, 1-5/8"; #2, 6-1/3"; #3, 10-5/8"; #4, 15-1/8"; #5, 19-5/8"

***Denotes different sample lot.

****Five velocity stations.

Table III-13 gives measurements of "detonation velocity" reported by Cruice[36] for several liquid gun propellant fuels, oxidizers, and mixtures. The test procedure is another modification of the card gap test. The primary test result is the propagation velocity of the shock wave through the sample material which is induced by an explosive donor. The material is contained in a section of schedule 80 stainless steel tubing (2 inch ID) 8 inches long. The tube bottom is sealed with a thin plastic diaphragm. The 160 grams RDX donor is placed directly below the diaphragm. A cold-rolled steel plate 4" x 4" x 3/8" thick placed on top of the sample container serves as a witness plate. The container is equipped with a constant current resistance wire circuit for measurement of the reaction wave velocity.

Table III-13. Detonation Velocity Test Results[36]

Material	Detonation Velocity (km/sec)	Remarks
2.8 Molar HAN	1.83	Tube in strips, plate OK, no detonation
2.8 Molar HAN	1.87	Tube in strips, plate OK, no detonation
11 Molar HAN	2.21	Tube in strips, plate OK, no detonation
11 Molar HAN	2.15	Tube in strips, plate OK, no detonation
13 Molar HAN	2.70	Tube in strips, plate OK, no detonation
13 Molar HAN	--	Tube in strips, plate OK, no detonation
NOS-365	2.63	Moderate tube fragmentation, plate bowed, LVD*
NOS-365	3.05	Moderate tube fragmentation, plate bowed, LVD*
LGP-1776	2.35	Moderate tube fragmentation, plate broken, LVD
LGP-1776	2.49	Moderate tube fragmentation, plate bowed, LVD
LGP-1845	2.56	High tube fragmentation, plate broken, LVD
LGP-1845	2.35	High tube fragmentation, plate broken, LVD

*Low velocity detonation

2. Impedance Mirror. Table III-14 gives reaction times (time for passage of the reaction zone) reported by Mallory[50] using the impedance mirror test described in Section II.

50. H. Mallory, "Function and Safety Tests of NOS-365 Monopropell[illegible], Naval Weapons Center NWCTP 5940, China Lake, CA, April 1977.

Table III-14. Reaction Times Measured with the Impedance Mirror Test

Material	Reaction Time (μsec)
NOS-365	~ 10.0
NM	0.22 ± 0.03
NM/Acetone 75/25	0.4

3. Heavy Confinement Shock Tests. Shock sensitivity tests of liquid gun propellants and torpedo fuels confined by heavy wall tubes have been reported by Mallory[50] and by Mason et al.[12]

a. Mallory reported tests of NOS-365 and nitromethane in heavy wall tubes as follows. A mild steel tube, 25 mm ID, 25 mm wall thickness and 0.9 m length was closed at one end with a welded steel plug. An RP-81 exploding bridgewire detonator was lowered to the bottom (closed end) and the tube was filled with the test liquid. The tube top was open. Figure III-6 shows the fragmented steel tube which contained NOS-365 at 15-21°C, and Figure III-7 shows the fragmented steel tube which contained 13-molar HAN solution at approximately 15°C. Mallory stated the fragments of the tube in both tests indicated brittle fracture patterns frequently observed in ruptured pressure vessels failing at low pressures; no measurements were made to determine whether detonation velocities were achieved. A 0.76 m length of 4340 steel Mann barrel, 20 mm ID and 19 mm wall thickness, was closed at one end with a welded steel plug. The barrel which contained a 2.5 gram tetryl pellet attached to an RP-81 detonator was filled with the test liquid. Figure III-8a and III-8b show the Mann barrel which contained NOS-365, indicating failure at a distance of about 12-15 in. from the booster; and Figure III-9a and III-9b show barrel fragments from tests with nitromethane and composition C-4 respectively.

b. Mason, Ribovich, and Weiss[12] reported tests of OTTO-II torpedo fuel with the confinement test described in Section II. The test result is shown in Figure III-10 which indicates complete fragmentation of the tube and holing of the witness plate.

D. Miscellaneous Tests

1. Cap Sensitivity. Smith and Harrison[38] reported a negative response to J-2 Cap Sensitivity Tests for OXSOL-I (3 trials) and OXSOL-II (5 trials). Kirshner and Silverstein[42] reported negative response to No. 8 Cap Sensitivity Tests for OTTO-II fuel.

2. Trauzl Block. Kirshner and Silverstein[42] reported that hydrazine-hydrazine nitrate water mixtures with more than 6% water gave only "partial" or low order detonation response in the trauzl block test.

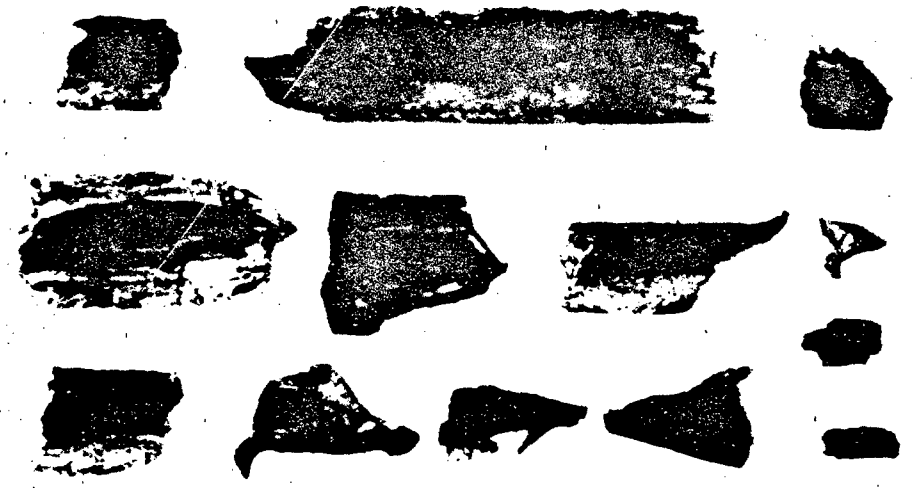
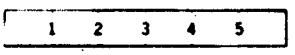


Figure III-6. Tube Test Fragments of Mild Steel from Detonation of NOS-365[50]



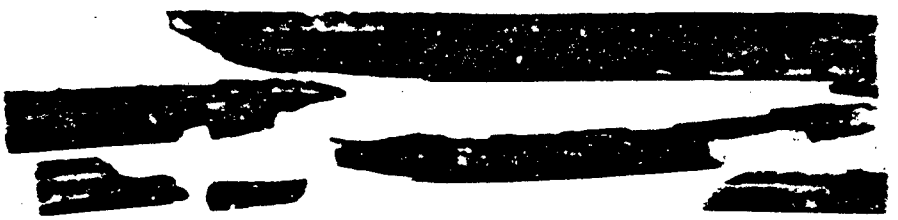
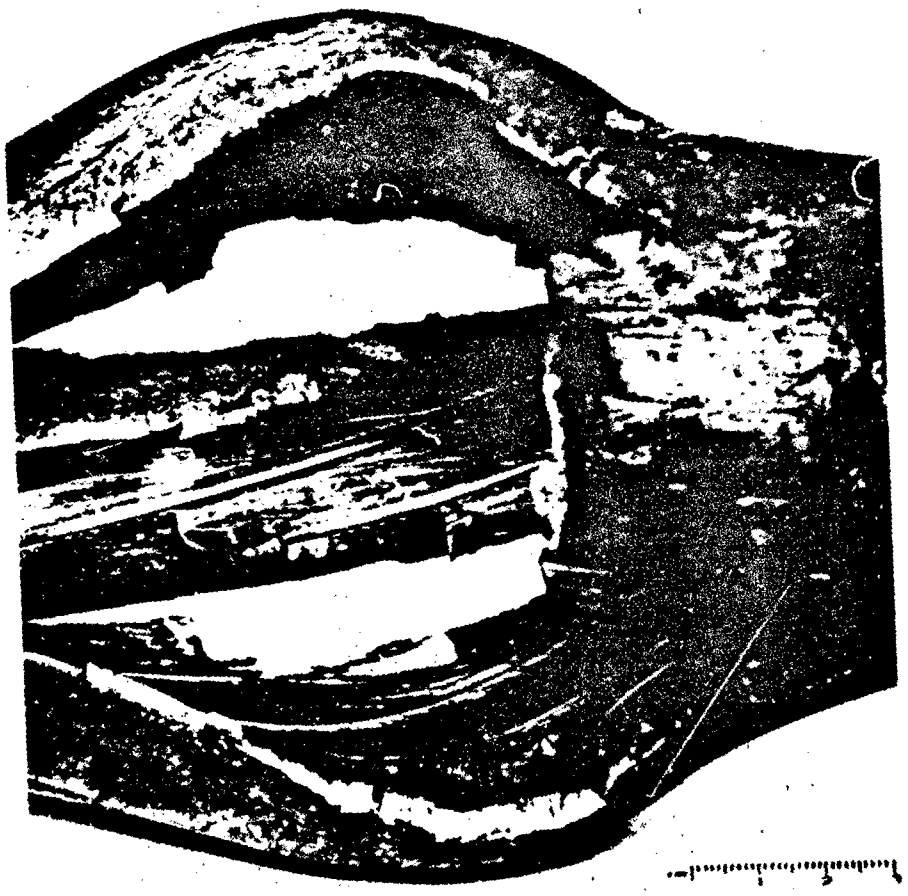


Figure III-7. Tube Test Fragments of Mild Steel from Detonating 13 Molar HAN Solution[50]

a.

b.



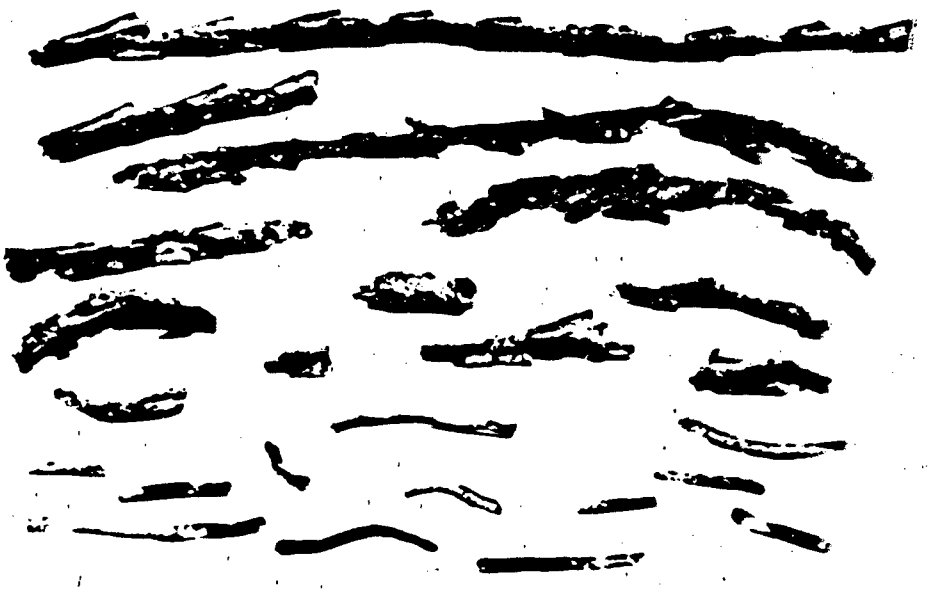
Closeup of detonation section

Figure III-8. Section of 20 mm Mann Barrel Used in NOS-365 Test[50]

a.

Nitromethane

b.



Composition C-4

Figure III-9. 20 mm Mann Barrel Fragments from Tests on NM and Composition C-4[50]

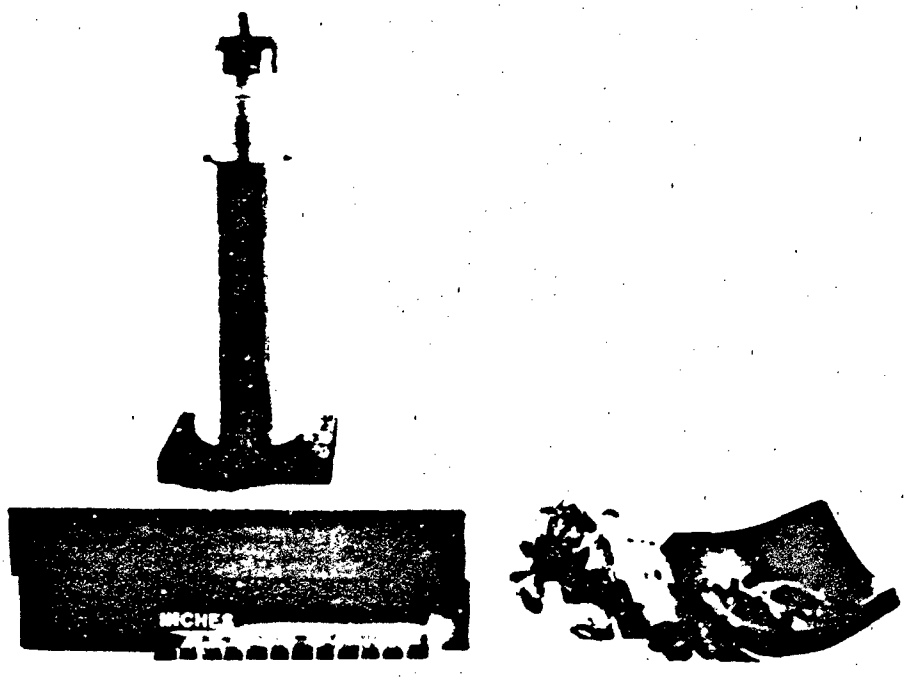


Figure III-10. Confinement Test Results for OTTO-II

Table III-15 gives Trauzl Block Test results for several liquid gun propellant fuels, oxidizers and mixtures.

Table III-15. Trauzl Block Test Results[36]

Material	V cc/gram		
	1 gm Load	2 gm Load	3 gm Load
2.8 M HAN	1.0	1.0	1.0
11 M HAN	4.4	3.0	3.7
13 M HAN	5.0	4.0	4.5
IPAN	0.5	1.0	0.7
TMAN	0.6	1.2	0.9
TEAN	3.0	1.5	2.7
NOS-365	4.0	3.2	3.6
LGP-1776	6.5	3.0	4.7
LGP-1845	6.0	4.2	5.1

3. Bullet Impact. Table III-16 gives reported bullet impact test results for several liquid gun propellants and some other energetic liquids which are included for comparison. These data are included in the "miscellaneous" test category rather than in the section on impact

sensitivity due to the relatively poor test condition definition and the fact that the test is directed to the containment of the liquid.

Table III-16. Bullet Impact Test Data

Material	Test Condition	Result	Reference
NOS-58	No details given	Positive (explosion)	40
NOS-283	0.50 caliber	No detonation, no fire/ 5 tests	38
NOS-283	0.30 caliber	No detonation, no fire/ 20 tests	38
NOS-365	0.50 caliber	No detonation, no fire/ 2 tests	38
OTTO-II	No details given	Negative (burned)	40
OXSOL-I	No details given	Negative/4 tests	37
OXSOL-II	No details given	Negative/4 tests	37
NPN	A-25-I*	3 explosions/3 tests	51
NPN	A-25-HE	3 explosions/3 tests	51
NPN	A-95-I	3 explosions/3 tests	51
NPN	A-95-HE	3 explosions/3 tests	51
NPN	A-95-.30 cal	No explosion (burned quickly)	51
H	A-25-I	No explosion/3 tests	51
H	A-25-HE	1 small explosion/3 tests	51
H	A-95-I	No explosion/3 tests	51
H	A-95-HE	Explosion/3 tests	51
H	A-95-.30 cal	No explosion or flames	51
HN Sol'n	A-25-I	No explosion/2 tests	51
HN Sol'n	A-25-HE	2 explosions/3 tests	51
HN Sol'n	A-95-I	No explosion/3 tests	51
HN Sol'n	A-95-HE	2 explosions/3 tests	51
EO	A-25-I, HE	Ignited, orange fireball	51
EO	A-95-I, HE	(every test/ 12 tests)	51
Aviation Gasoline (115/145)	A-25-I	Fire (2 out of 3 tests)	51
(115/145)	A-25-HE	No flames or explosion	51
(115/145)	A-95-I	Fire (2 out of 3 tests)	51
(115/145)	A-95-HE	Fire (1 out of 3 tests)	51

*A-B-C
where A = aluminum container, 6061, wall thickness = 0.064 in.
B = % liquid filled
C = ammunition (I = 20 mm incendiary, HE = 20 mm high explosive)

51. G. Glatts, "Stability Tests of Monopropellants Exposed to Flames and Rifle Fire," Jet Propulsion Laboratory Technical Report No. 32-172, California Institute of Technology, Pasadena, CA, February 26, 1962.

4. Bonfire and Unconfined Burning. Glatts[51] reported qualitative observations of response of hydrazine, hydrazine nitrate solution, normal propyl nitrate, ethylene oxide, and aviation gasoline (for comparison) in one gallon aluminum cans to wood and oil fire exposure. Explosions of varying intensity (and time to explosion) were observed for all these materials. Romero[39] reported that NOS-365 in a one gallon plastic container in a full metal shipping container with fiberglass packing did not explode in a wood bonfire but did ignite and burn quickly (30 seconds). Smith and Harrison[37] cited negative results (presumably meaning no explosion, but no details were given) for OXSOL-I and OXSOL-II in an "unconfined" burning tests. Cziesla et al.[40] reported that OTTO-II did not detonate in an unconfined burning test but that NOS-283 did detonate in the same test.

IV. DISCUSSION AND RECOMMENDATIONS

The purpose of this study was to review the hazardous material safety testing methods which have been applied to energetic liquids and to provide recommendations for their use, along with additional tests which might be indicated, for the assessment of liquid gun propellant reactivity (explosivity) hazards.

In Section II a rationale for development of a protocol for safety testing of liquid propellants is suggested. Test procedures are required which will quantify the response of the propellant to energy input conditions which may be experienced in handling, storage, and transportation. It is desired that the propellant should not react violently to such energy input conditions. Since the propellant must react with rapid, but controllable, energy release when ignited in the gun barrel, it is required that the differences in conditions experienced by the propellant in the gun application and in storage, handling, and transportation be quantified. Such conditions include thermal energy (heat) inputs and energy inputs to the material by localized compression, such as might be experienced due to impact in a transportation accident or in the pre-ignition pressurization of propellant during gun loading. Thermal explosion theory suggests the response of a propellant to energy inputs depends not only on the rate and magnitude of the energy input but also on other initial and boundary conditions imposed on the material. It follows that the response of the propellant to energy inputs can depend on the material's confinement and on its initial state (such as temperature or presence of air or vapor bubbles).

Current safety testing procedures for energetic liquids, described in Section II, rely on comparison of the responses of different materials to a particular energy input condition. Most of the tests described were designed to provide a purely relative measure of the sensitivity of energetic liquids to the selected test condition rather than to provide information which could be used to predict the responses of a specific material to more general conditions. Further, the test conditions appear to have been selected with primary orientation to the identification of hazardous responses which might be experienced in the propellant end use application. For example, a measure of the propellant's propensity for detonation (and its prevention via propellant application equipment design) appears to have been a primary focus for the test procedures.

The application of the described test methods to assessment of potential hazards associated with liquid gun propellants in storage, handling, and transportation is difficult for two primary reasons: First, the conditions which characterize the normal and accident transportation, storage, and handling "environment," i.e. the characteristics of the energy inputs that can be expected, are not known. This problem, although long recognized, has received little attention, even though such information is prerequisite to the specification of test conditions to be applied in safety test protocols. Second, the individual test procedures, each addressing only one specific energy input condition, can provide only one input to an energy input-material response matrix which would encompass the conditions anticipated in storage, handling, and transportation. Unless the individual

test results are considered with results from other tests which provide additional information about the material's response to conditions that in some meaningful way imitates those which may be experienced in handling, storage, and transportation, their application to determining the safety of a material can be difficult, if not misleading. Furthermore some of the tests involve fairly subjective "go/no-go" results, i.e. the standard card gap and drop weight tests which involve observation of mechanical damage of a particular degree (witness plate holing or sample container failure). These factors make it difficult to compare sensitivity "between tests" and, in some cases, to provide quantitative ordering of sensitivity of different materials to the same test.

From this survey it appears that the characterization of the sensitivity of a material to explosive energy release during storage, handling,and transportation inevitably requires a number of tests which provide information about the material's response to different energy input conditions. In this regard the study reported by Cruice,[36] which describes the application of a battery of tests to determine the sensitivity of liquids for hazard classification for shipping provides information for safety assessment which cannot be obtained from any single test procedure. However it appears that the test battery performed by Cruice was specified primarily to provide a reasonably close correlation with the test protocol used for military solid explosives as described in Department of the Army Technical Bulletin C1, TB 700-2; and it is not clear how well such a test protocol relates to the conditions to be encountered in storage, handling,and transportation. The classification proposed by Cruice addresses two types of energy inputs: thermal and impact/shock. It is probable that the use of a battery of tests such as proposed by Cruice for determining sensitivity to thermal energy inputs, which includes (a) Flash Point and Ignition Temperature determination and (b) thermal stability determination including isothermal, long term exposure and programmed temperature studies, with provision for observation of confinement and container catalysis effects, provides sufficient information on sensitivity to thermal stimuli to allow confident ordering of the thermal energy input sensitivity of liquid propellants for safety evaluation purposes. However, it is less clear how well the test procedures for impact/shock energy input provide information for assessment of potential hazards in storage,handling,and transportation. Such judgments can only come from comparison of test results for gun propellants with those obtained for other materials for which there is a history of satisfactory performance in the storage, handling,and transportation environment. Consequently, the results of a battery of tests on liquid gun propellants would be more useful for hazard evaluation if the test battery were also applied to other energetic liquids such as nitromethane and nitroglycerine for comparison. It also appears that certain kinds of conditions which might lead to low velocity detonation of some of the propellants might not be sufficiently delineated in a test protocol such as the one suggested by Cruice.

Aside from the problem of definition of the conditions to be tested for, i.e. the range of conditions which the propellant will be exposed to in the transportation, handling,and storage environment, the data available on the response of liquid gun propellants to the tests described in Section II is fragmentary and not sufficient for the evaluation of the test's usefulness for delineation of liquid gun propellant hazards. However, it is

instructive to attempt a sensitivity ranking of liquid gun propellant materials using the data available from the "standard" impact and shock energy input tests.

Table IV.1 gives results of card gap, drop weight, adiabatic compression, and low amplitude compression wave tests, summarized from Section III, for several liquid propellants. Table IV.2 gives a ranking for the materials for each test based on the data of Table IV.1. Lower numbers denote higher "sensitivity" as measured by the test result. The materials shown are ranked similarly by the drop weight, card gap, and adiabatic sensitivity tests. However, a different ranking is obtained based on the results of the low-amplitude compression wave test. The similar indications of "sensitivity" for OTTO-II, NOS-365,and NM obtained from the low amplitude compression wave test, which is in contrast to the indicated order of sensitivity for these materials based on the drop weight, card gap, and adiabatic compression tests, as well as from measurements of reaction rates (times) such as those presented by Mallory,[50] are important to understand for purposes of safety evaluation. This difference in ordering is not surprising, in view of the wide differences in the energy input magnitude and rates and in the boundary conditions imposed on the material in the different tests. It may be that the low amplitude compression wave test result is indicative of a similar propensity for low velocity detonation of these materials under that specific test condition and the other three tests may not (probably do not) measure this propensity. As has been noted by Watson, the low amplitude compression wave test indicates a similar propensity for explosive energy release (probably best characterized as LVD) that would not be indicated for NOS-365, OTTO-II,and NM from the other three tests. It has been suggested that the conditions experienced in the low amplitude compression wave test may better imitate the conditions experienced in some transportation accidents.

Table IV-1. Selected Test Results for Liquid Gun Propellants

Material	Drop Weight (kg-cm)	Results for Adiabatic Compression (kg-cm/ml)	Low Amplitude Compression Threshold Velocity (m/sec)	NOL Card Gap (mils)
NOS-365	152	--	26.2	0
OTTO-II	8.5-34.2	7.6-21.8	23.4	10-150
H	>200	--	>76	0
PN	15.5	4.6-6.7	91.3	--
EN/PN	5.8	4.0	--	100
NM	37.3	10.4	24.1	150-300
NG	2.5	--	--	380-500

Table IV-2. Sensitivity Order of Test Results from Table IV-1

Test	NOS-365	OTTO-II	H	PN	EN/PN	NM	NG
Drop Weight	6	4	7	3	2	5	1
Adiabatic Compression	--	4	--	2	1	3	--
Low Amplitude Compression Wave	1	1	2	3	--	1	--
NOL Card Gap	5	4	5	--	3	2	1

Additional work is required to provide a better data base for predicting and assessing the hazard potential of liquid gun propellants, and the following recommendations are offered.

1. A basic program should be undertaken to provide test results for the liquid gun propellants of current interest, as well as a number of other energetic liquids (such as nitromethane, ethylene oxide, nitroglycerine, for which there is an extensive "experience" data base) using the instrumented Card Gap Test, the drop weight and adiabatic sensitivity tests, the thermal surge test, and the low amplitude compression wave test. This requirement is necessitated by the current lack of data on these materials which can be meaningfully compared. The proposed exercise is similar to that performed by Cruice[36] but would include additional test procedures and other energetic liquids for comparison.

2. A parallel effort should be made to characterize the input energy and boundary conditions for each of these tests to provide a means for evaluating the tests with respect to "severity" and correspondence to conditions which may be encountered in handling, storage, and transportation. Some work along these lines, directed to characterization of the drop weight, adiabatic sensitivity, and card gap tests, has been done by the Bureau of Mines. It is expected that such an effort would indicate that some tests now performed are redundant or not applicable to liquid gun propellant safety evaluation.

3. A series of tests utilizing the card gap principle should be run to determine the effect of confinement on the propensity for low velocity and high velocity detonation. These tests should be instrumented to obtain continuous reaction front velocity and pressure as a function of distance. Presence of gas phase (bubbles), degree of confinement, and reaction time (length of reaction path) should be test variables. This test series should provide information on the propensity for low velocity detonation. These tests would be similar to those conducted by Cruice,[36] Pulsepower Systems, Inc.,[49] and the Bureau of Mines[35] but would provide for additional test variables and would be carried out for a number of other energetic liquids for which there is good "experience" data.

4. The compression sensitivity tests as conducted by Princeton Combustion Research Laboratories[46,47,48] should be conducted for the other liquid gun propellants of interest and for other selected energetic liquids for comparison. Although these tests were designed to provide information

on safe gun operating conditions, they should also be useful in determining the relative sensitivity of gun propellants compared to other energetic liquids for which there is a better experience data base.

5. Methods should be explored for acquiring more fundamental data on the energetics and kinetics of reaction of liquid propellants. Measurement of global rate and activation energy constants, using techniques such as accelerating rate calorimetry should be evaluated. Data from constant volume propellant burning tests conducted at the Ballistic Research Laboratory[52,53] (which were not reviewed in this work) should be evaluated for their application to the identification of sharp pressure transitions due to changes in the mode of combustion, as observed for NOS-365 propellant. The latter may be important in the determination of design criteria for safe containers.

6. Finally, it must be emphasized that the development of test protocols must take into account the anticipated practices regarding container types (which will determine confinement and chemical compatibility effects) and sizes (which may determine inertial impact effects as well as (self) confinement).

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GLOSSARY

AN	ammonium nitrate
AP	ammonium perchlorate
BEN	benzene
DNG	glycerol dinitrate
DNP	dinitroxypropane
ED	ethylene diamine
EG	ethylene glycol
EGDN	ethylene glycol dinitrate
EGMN	ethylene glycol mononitrate
EN	ethyl nitrate
EO	ethylene oxide
H	hydrazine
HAN	hydroxyl ammonium nitrate
HN	hydrazine nitrate
IPAN	isopropyl ammonium nitrate
IPN	isopropyl nitrate
LGP	"liquid gun propellant"
MMAN	monomethyl ammonium nitrate
MMH	monomethylhydrazine
MNG	glycerol mononitrate
NDPA	nitrodiphenylamine
NEN	normal ethyl nitrate
NG	nitroglycerine
NM	nitromethane
nos	"not otherwise specified"
NP	nitropropane
NPN	normal propyl nitrate
OCT	octane
PDDN	dinitroxy propane
PN	propyl nitrate
RDX	cyclotrimethylenetrinitramine
TEAN	triethanolammonium nitrate

TEGDN	triethylene glycol dinitrate
TMAN	trimethylammonium nitrate
TMETN	trimethylolethane trinitrate
TNM	tetranitromethane
UDMH	unsymmetrical dimethyl hydrazine
WFNA	white fuming nitric acid

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